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ABSTRACT

This manual was developed for use at workshops designed to upgrade the knowledge of experienced wastewater treatment plant operators. Each of the lessons in this document has clearly stated behavioral objectives to tell the trainee what he should know or do after completing that topic. Areas covered in this manual include: types and factors affecting activated sludge processes, identification and solution of operating problems, selected tests and measurements, and chemical determinations. A glossary of terms is included for reference. (CS)

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ED155003

ACTIVATED SLUDGE PROCESS WORKSHOP MANUAL

1976

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ACTIVATED SLUDGE PROCESS

WORKSHOP MANUAL

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INTRODUCTION

The *Activated Sludge Process Workshop* manual was developed jointly by the Wastewater Technology Section, Pollution Control Branch, and the Training and Certification Section. It has been prepared as a home study and reference manual for plant operators and as the text for the related workshop.

The principal objective of the Workshop is to upgrade the knowledge of experienced wastewater treatment plant operators. The lesson objectives are clearly indicated at the beginning of each topic and tell the operator what he must know or do after having covered the topic. Upon completion of the Workshop, the trainee is expected to attain a minimum level of competence of 70% for the course.

This is a working course in which each person is expected to take an active part in subject discussions. It includes considerable hands-on participation, in order to provide as much practical knowledge as possible of activated sludge process control.

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SUBJECT: 1

ACTIVATED SLUDGE PROCESS

TOPIC: 1

TYPES OF PROCESSES

OBJECTIVES:

The trainee will be able to

1. Recall the principle of the Activated Sludge Process.

2. Describe verbally and/or using a diagram the conventional activated sludge process.

3. Name and differentiate between the three types of activated sludge processes.

4. Recall:

   a) the normal BOD_5 for raw sanitary sewage.

   b) the reduction of BOD achieved by primary treatment.

   c) the reduction of BOD achieved by both primary and secondary treatment.

5. Define terms used in the Activated Sludge Process such as:

   a) BOD

   b) Mixed Liquor

   c) Nitrification

   d) Aerobic

   e) Coagulation

   f) Flocculation

ACTIVATED SLUDGE

OPERATING PRINCIPLE AND TYPES OF PROCESSES

BIOLOGICAL OXIDATION

Wastewater, if it is biodegradable, can be reduced biologically by reactions similar to those that occur naturally in soils and surface waters. The activated sludge process concentrates these reactions under more controlled conditions by maintaining large numbers of aerobic bacteria (supported by oxygen). These bacteria use the components of wastewater whether domestic or industrial as a food source and also take part in biochemical reactions that convert the soluble and colloidal material portion to a disposable insoluble material (floc).

To facilitate these reactions raw or settled wastewater (primary effluent) is added to an aeration basin where air is provided and a floc or mixed liquor is developed through the oxidation of organic substances. The mixed liquor is then concentrated by sedimentation in a clarifier. The thickened mixed liquor or sludge is returned to the aeration basin to maintain a population of bacteria there and the clarified supernatant is chlorinated for disinfection and discharged to a receiving stream.

Portions of settled mixed liquor must be wasted from the system to prevent overloading as the wastewater is utilized and sludge growth continually takes place. This portion is classified as waste activated sludge and is treated further in a digester.

CARBONACEOUS BOD

The strength of wastewater is often measured by the five-day Biochemical Oxygen Demand (BOD_5) test which is the amount of oxygen required (mg/l) by the process bacteria in utilizing organic carbon compounds (carbohydrates) over a five-day period. For this reason the BOD_5 is often referred to as the carbonaceous oxygen demand. The oxidation of the

organic carbon compounds by bacterial action produces carbon dioxide which is evolved as a gas, thus carbon removal is accomplished. These reactions are illustrated as follows:

$$\underset{(BOD_5)}{\text{carbon}} + \text{oxygen} \xrightarrow{\text{bacteria}} \text{carbon dioxide} \uparrow$$

NITROGENOUS BOD

If the process sludge is held within the system long enough and sufficient oxygen is provided, a further reaction will occur called Nitrification. Under these conditions, nitrogen bacteria will populate the aeration basin and oxidation of ammonia and some organic nitrogen as well as carbon compounds will occur. Since nitrogen compounds also impose an oxygen demand they are referred to as the nitrogenous oxygen demand (NOD). These reactions involve the biological oxidation of organic and ammonia nitrogen to nitrite and nitrate and are illustrated as follows:

$$\underset{\text{(ammonia \& organic)}}{\text{nitrogen}} + \text{oxygen} \xrightarrow{\text{bacteria}} \text{nitrite} + \text{nitrate}$$

Bacteria require phosphorus as well as nitrogen and carbon for their metabolism; therefore some phosphorus is removed from the wastewater as well in the activated sludge process. Unfortunately, only a small quantity of phosphorus is needed by the bacteria and consequently only minimal phosphorus removal is attained.

In general the activated sludge process can be stated as being a carbon (BOD_5) removal process with the possibility of nitrogen conversion as well. (See Figure 1-1). Phosphorus removal (particularly soluble) must be attained by chemical addition to the process. See Topic 6.

Not all the reactions in an activated sludge process are chemical in nature. Adsorption of particulate matter on to the floc is also attained in the aeration basin.

In summary, biological purification occurs in the Activated Sludge Process in the following matter:

1. Coagulation and flocculation of soluble and colloidal matter.
2. Oxidation of carbonaceous compounds.
3. When process conditions are adequate oxidation of nitrogenous compounds.

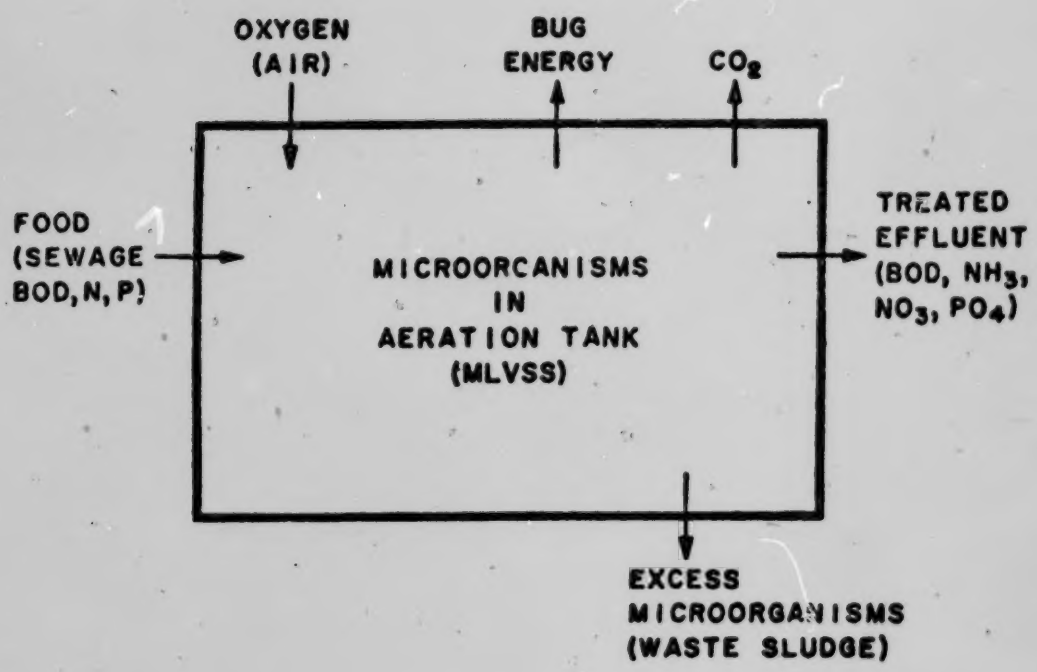


Figure 1-1 BIOLOGICAL TREATMENT PROCESS

TYPES OF ACTIVATED SLUDGE PROCESSES

The different types of Activated Sludge Systems include:

1. The conventional Activated Sludge Process (Figures 1-2 and 1-3).
2. Contact stabilization (Figures 1-4 and 1-5).
3. Extended Aeration (Figures 1-6 and 1-7).
4. Step aeration.
5. High-Rate.

These systems operate on the same biological principles and usually feature similar microbial populations. They differ by operating at specific loading rates and employing specific physical plant designs.

The Conventional Activated Sludge System

This design employs primary clarifiers ahead of rectangular shaped aeration tanks to remove the easily settled solids in the influent wastewater. The solids are removed for further treatment usually in an anaerobic digester prior to ultimate disposal. The effluent from the primary clarifier, containing less organic matter, imposes a smaller load on the aeration section than would raw sewage. This makes smaller aeration tanks possible, and produces a lower demand for oxygen. Aeration Detention time is usually 6 to 8 hours.

The activated sludge (mixed liquor) developed in the aeration basin is in turn settled in the secondary clarifier. Waste activated sludge is directed to the primary clarifier or a sludge thickening tank and is digested along with the raw sludge from the primary section.

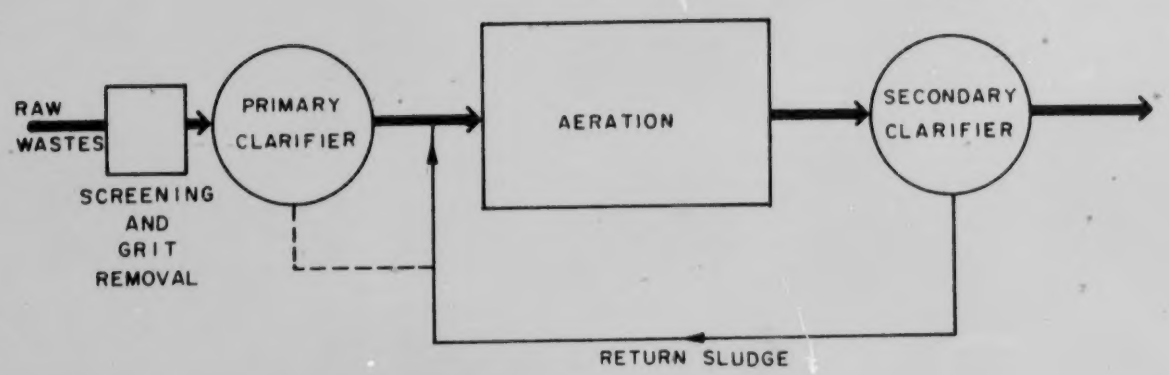


Figure 1-2 CONVENTIONAL ACTIVATED SLUDGE SYSTEM

The conventional activated sludge process is capable of removing 85 to 95% of the incoming wastewater BOD_5 and produces an effluent suspended solids of less than 20 mg/l. Varying amounts of ammonia, nitrite and nitrate will appear in the secondary effluent depending on the extent of nitrogenous oxidation in the aeration basin.

Normally the sludge produced by this process is 0.5 to 0.6 lbs/lb BOD_5 reduced for domestic wastewater; the sludge yield on industrial waste can be lower or higher than this value.

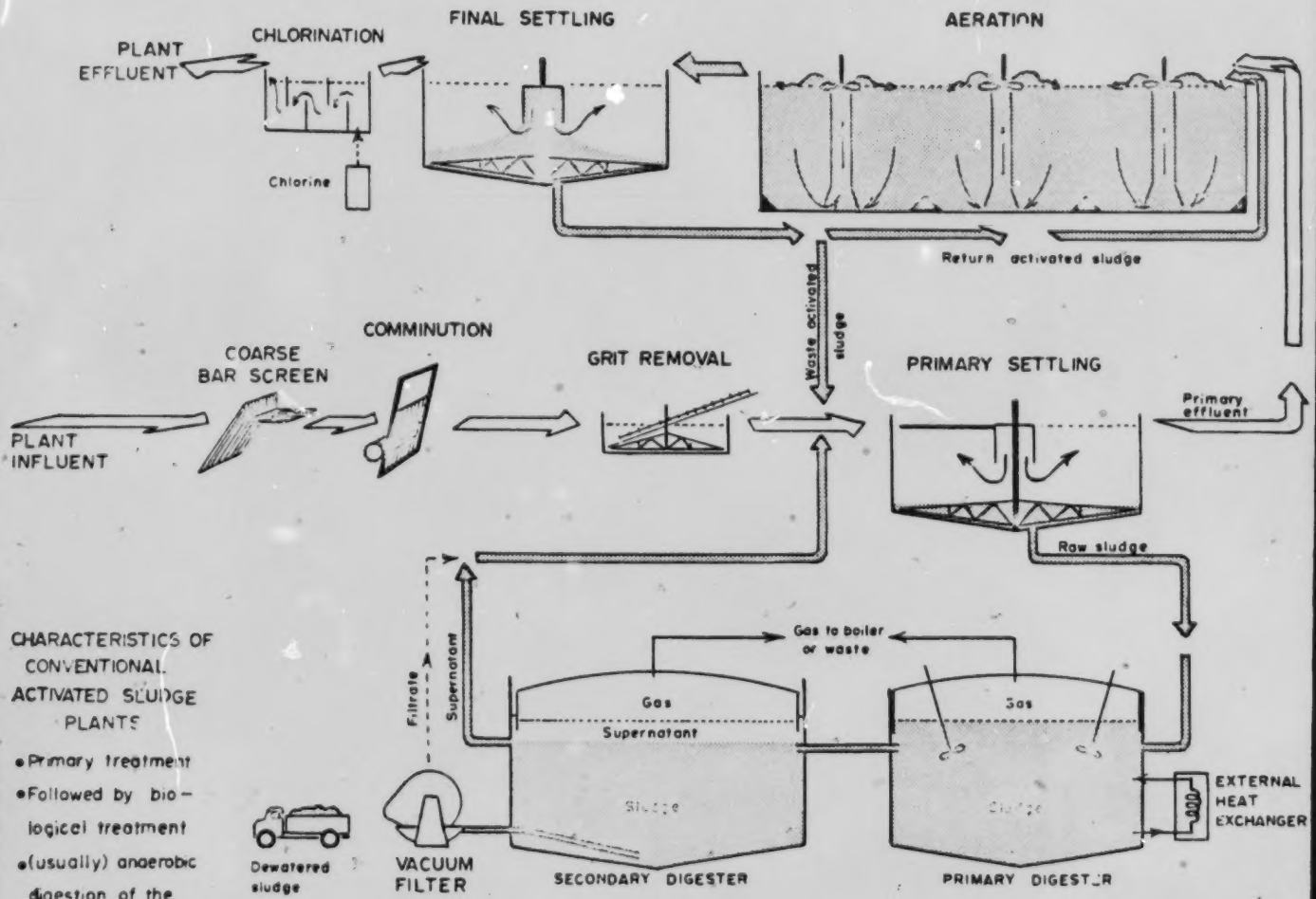
Contact Stabilization System

If a large part of the wastewater is in colloidal or suspended form, this system provides a means of varying the contact time to achieve the required reduction in the influent BOD. Wastewater, with no primary sedimentation, is mixed in the contact aeration tank with stabilized activated sludge. The period in the contact aeration tank is usually about 1-2 hours. During this short detention period the suspended particles are adsorbed on the floc. The floc is then separated from the bulk of the liquid by settling in the clarifier. The settled sludge is pumped from the clarifier to a tank (re-aeration tank) where it is further aerated for periods of 2-6 hours. Thus the total detention time in the system is 3-8 hours. This allows the microbes to assimilate the soluble adsorbed solids, producing a stabilized activated sludge for further treatment. Nitrification is usually not consistently achieved in this process arrangement, due to short hydraulic detention times in the contact aeration section.

Excess (waste) activated sludge is usually stabilized in an aerobic digester(s). The organic content of the waste sludge is reduced producing a non-putrescible sludge which is then disposed of. Sludge production in a contact process is stated as being 0.6 to 0.8 lb. solids/lb. BOD_5 reduced. The effluent quality is usually as good as that of a conventional process.

FIGURE No. 1-3 CONVENTIONAL ACTIVATED SLUDGE

Shown here with circular centre-fed clarifiers, mechanical aerators, two stage sludge digestion, and sludge dewatering by vacuum filtration.



TWO STAGE DIGESTION

CHARACTERISTICS OF CONVENTIONAL ACTIVATED SLUDGE PLANTS

- Primary treatment
- Followed by biological treatment
- (usually) anaerobic digestion of the mixed sludge

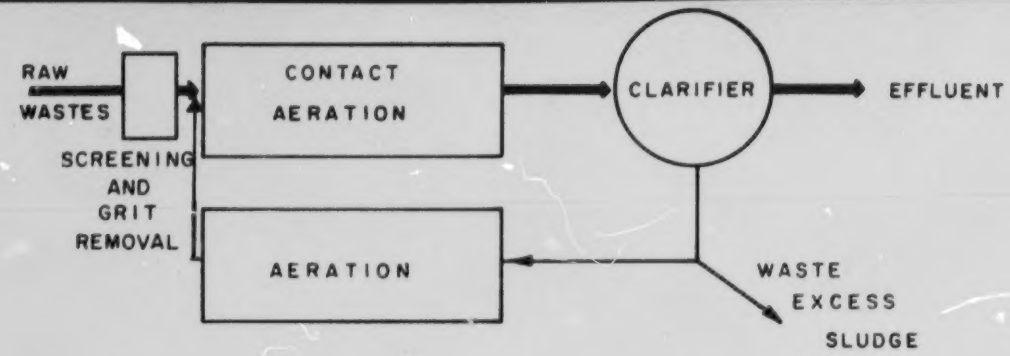


Figure 1-4 CONTACT STABILIZATION

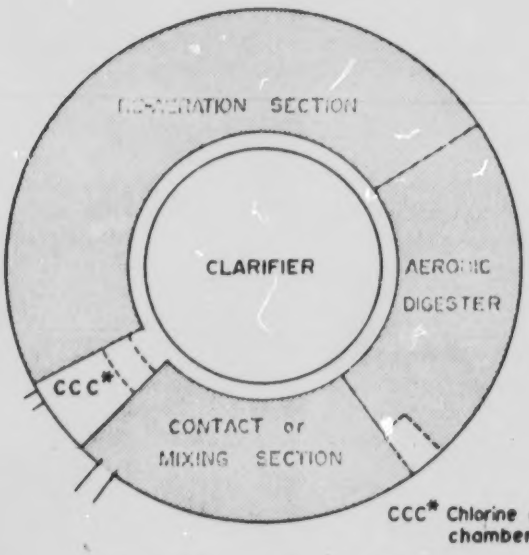
Extended Aeration System

The extended aeration process arrangement, formerly known as "total oxidation", involves longer periods of aeration detention; usually 18 to 24 hours for domestic wastewater treatment. Applications of this process for industrial wastewater treatment (e.g. textile industry) often exceed this detention time when a slow biodegradable material is being treated.

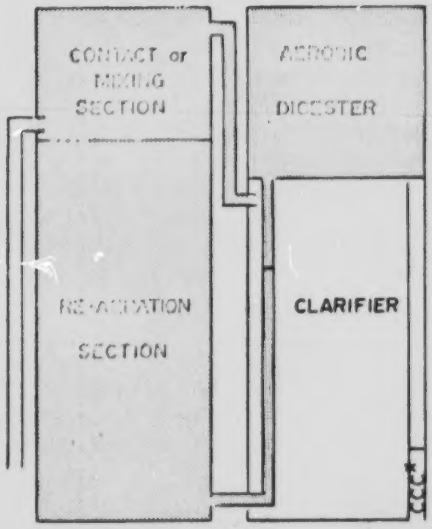
Due to these long aeration periods and resultant low process loadings, complete oxidation of carbonaceous and nitrogenous compounds, under endogenous respiration is attainable. Consequently, primary sedimentation is normally not employed and aerobic digestion is used for waste activated sludge stabilization.

The aeration section can be either of completely mixed, or plug-flow arrangement such as the oxidation ditch configuration.

The process sludge production is lower than in the conventional and contact process as 0.15 to 0.4 lb. solids/ lb. BOD_5 reduced is given.



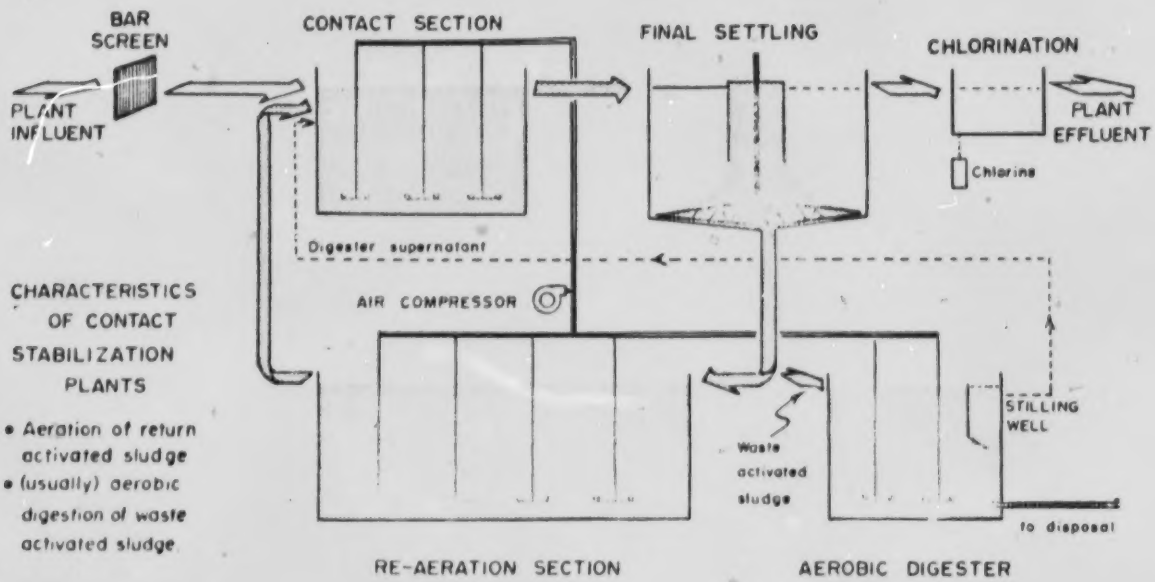
Components are arranged around a a central circular clarifier.



Components are arranged in a rectangular tank.

FIGURE No. 1-5 CONTACT STABILIZATION

Shown here without grit removal facilities since these are not normally supplied as part of the "package".



CHARACTERISTICS OF CONTACT STABILIZATION PLANTS

- Aeration of return activated sludge
- (usually) aerobic digestion of waste activated sludge

The effluent from an extended aeration process is usually low in BOD_5, achieving in excess of 90% reduction. Ammonia and organic nitrogen levels are also low due to total oxidation.

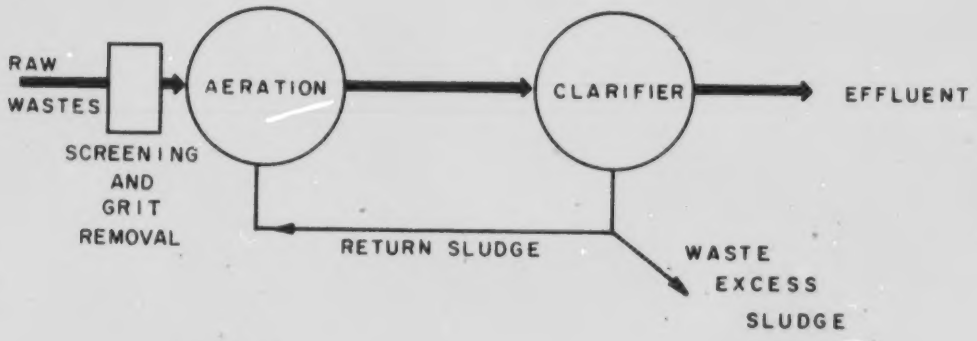


Figure 1-6 EXTENDED AERATION

Step Aeration System

This process design is often classified as another process type, but in fact it is just a modification of the conventional system. The difference is that primary effluent is fed in steps along the aeration tank, thereby distributing the waste loading more uniformly to the activated sludge mass.

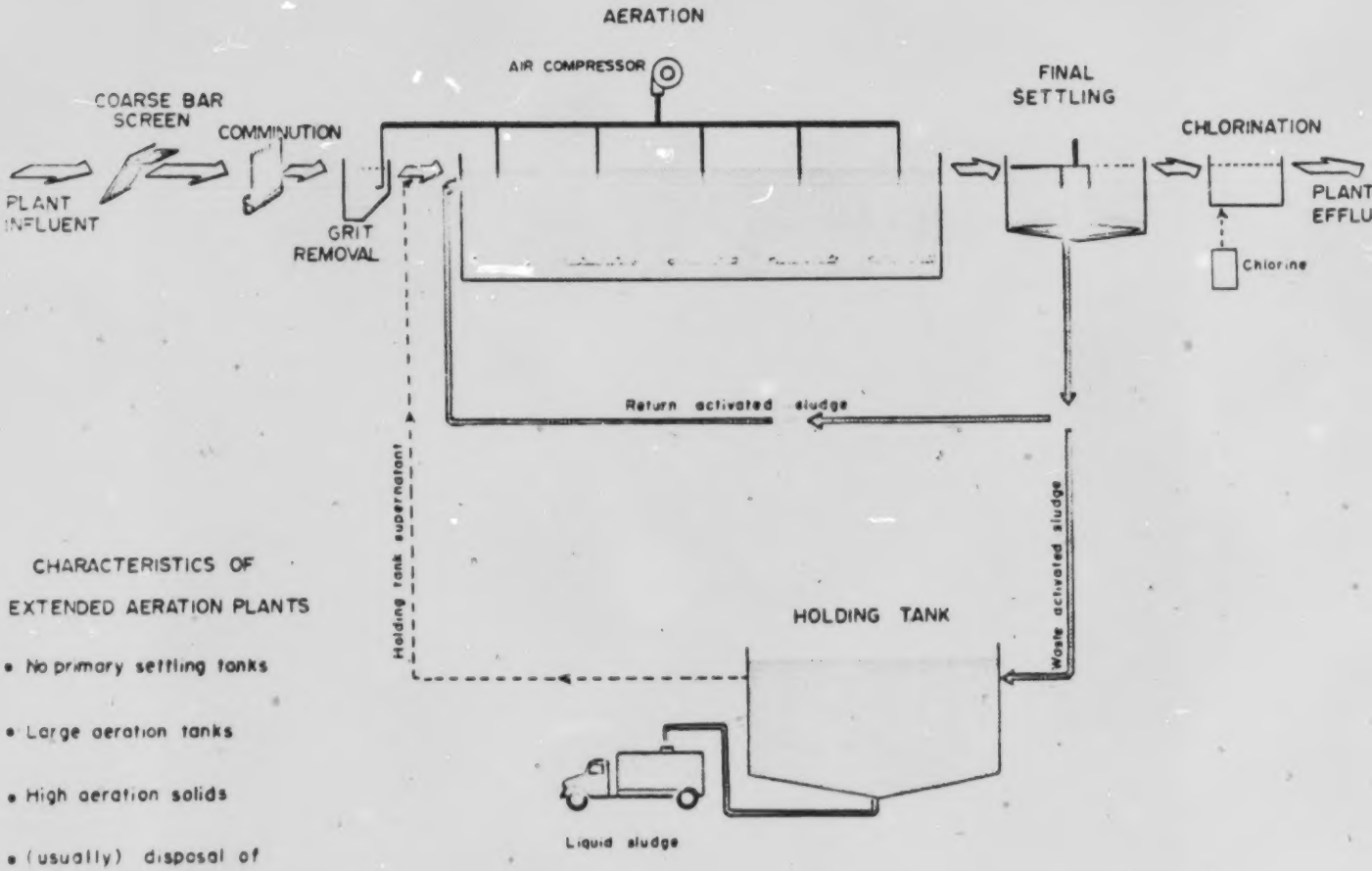
High-Rate Activated Sludge System

This process is used when only fractional wastewater treatment is required, such as an industrial waste discharge to a sewer system or in the first stage of a two-stage nitrification plant. With or without primary treatment, a completely mixed aeration basin having between 1 and 3 hours detention is utilized followed by final clarification and sludge return.

FIGURE No. 1-7

EXTENDED AERATION

Shown here with an aerated grit tank, aeration by diffused air, circular centre-fed clarifier, and a decanted sludge holding tank.



CHARACTERISTICS OF EXTENDED AERATION PLANTS

- No primary settling tanks
- Large aeration tanks
- High aeration solids
- (usually) disposal of raw waste activated sludge

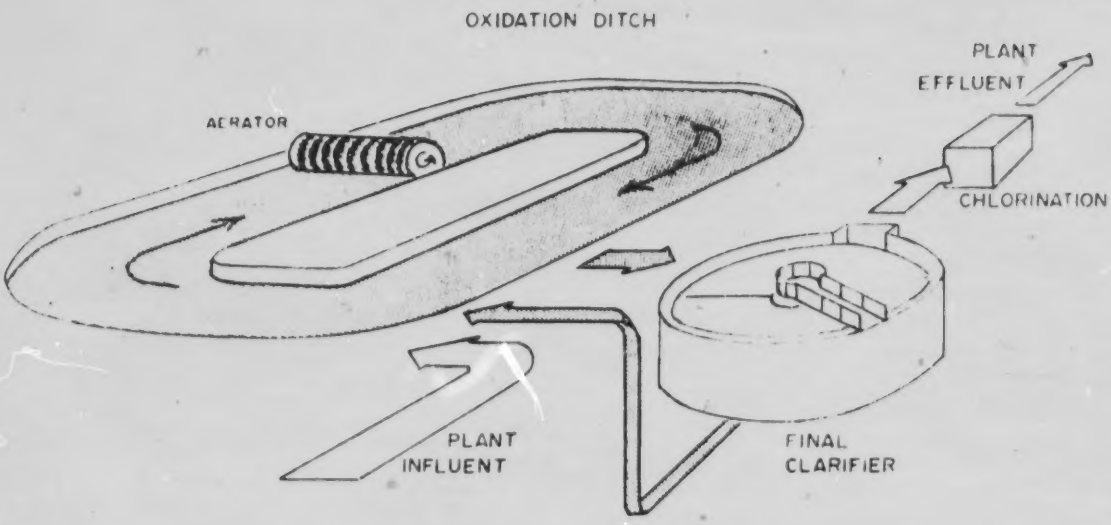
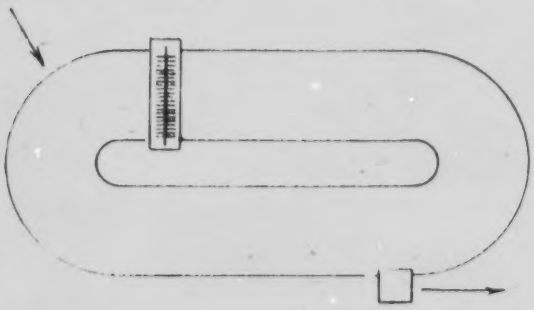
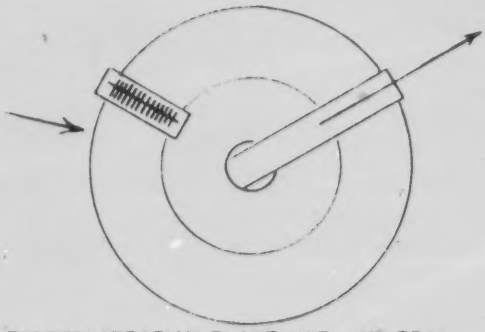


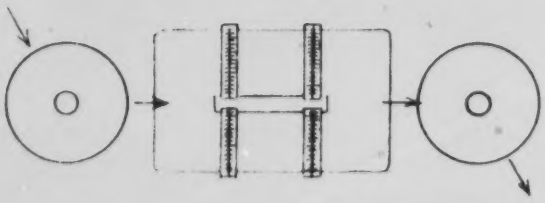
FIGURE 1-8 OXIDATION DITCH Shown above as an extended aeration plant. Other possible configurations are shown below.



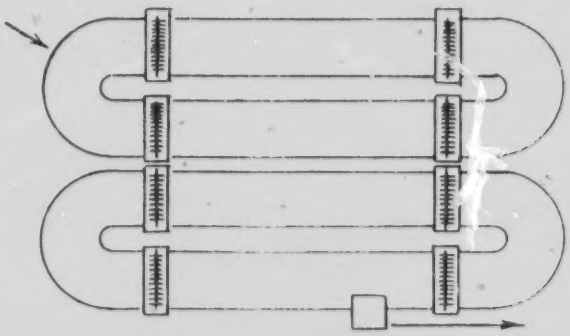
AERATED LAGOON



EXTENDED AERATION PLANT WITH CENTRAL CLARIFIER



CONVENTIONAL ACTIVATED SLUDGE PLANT



AERATED LAGOON WITH TWO OXIDATION DITCHES IN SERIES

SUBJECT:

ACTIVATED SLUDGE PROCESS

TOPIC: 2

FACTORS AFFECTING THE ACTIVATED SLUDGE PROCESS

OBJECTIVES:

The trainee will be able to

1. Name 8 factors that affect the process.
2. Identify those factors which can be controlled by the operator.
3. Identify the problems which may arise by changes in -
   a) Loading (Volumetric)
   b) F/M ratio
   c) Solids Retention Time
   d) Aeration D.O.
   e) Sludge Return Rate
   f) Temperature
   g) Aeration pH
   h) Industrial Waste
4. Recall:
   a) The characteristics of activated sludge.
   b) The normal waste loading of conventional and extended aeration plants.
   c) The optimum aeration basin pH range.
   d) The minimum aeration D.O. concentration required.

FACTORS AFFECTING ACTIVATED SLUDGE PROCESS

GENERAL

The ability of activated sludge to purify sewage will depend on its quality or condition; a good quality activated sludge is usually brown in colour and has a pleasant earthy odour, if it is kept well aerated. If deprived of oxygen for any length of time (9 to 12 hours) the sludge darkens and eventually becomes black, anaerobic and evil smelling. Characteristics of good and bad sludges are shown in Table 2-1.

Since the bacteria do the work in the activated sludge system, the factors affecting the system are those affecting the bacteria. As with all life forms, these organisms can only live if conditions remain suitable for their growth.

Factors which can affect the activated sludge process include:

1. Loading (Volumetric)
2. F/M ratio
3. Solids Retention Time
4. Aeration D.O.
5. Sludge Return Rate
6. Temperature
7. Aeration pH
8. Industrial Waste

TABLE 2-1 - GOOD SLUDGE vs BAD SLUDGE
(Domestic Wastewater)

A good sludge:	A bad sludge:
- will be coarsely granular	- will look light and fluffy - may take on gelatinous appearance
- will be chocolate to golden brown in colour	- can be gray in colour, or it can be exceptionally dark brown, shading to black
- will settle rapidly with little rivulets running through the sludge (usually more than 50% of its volume will settle in 5 minutes)	- will settle poorly
- will leave a clear sparkling supernatant	- will have a cloudy supernatant
The return sludge - If good, return sludge will: - have an earthy odour - be a chocolate brown colour	The return sludge - If under-aerated, sludge will: - have unpleasant, septic odour - be very dark

NOTE: The physical characteristics of activated sludges treating industrial wastes will vary depending on the type of wastes being treated, e.g. - milk waste activated sludges may be light brown to pinkish-red. Phenolic waste activated sludges may be dark brown to black in colour.

FACTORS

Loading (Volumetric)

Activated sludge process loadings are often expressed in terms of the weight of carbonaceous material (BOD_5) added to a 1,000 cu. ft. of aeration volume. For design purposes, the conventional and contact stabilization systems are loaded at 30 to 50 lbs BOD_5/1,000 cu.ft./day and the extended aeration system at 5 to 15 lbs BOD_5/1,000 cu.ft./day. Values exceeding 100 lbs. BOD_5/1,000 cu.ft. are normal for high-rate systems, but normally result a higher effluent BOD_5 with influent concentrations of nitrogenous matter.

Food/Microorganism Ratio (F/M)

The F/M ratio is a term used to describe the weight of wastewater BOD_5 applied to a weight of microorganism (MLVSS). This term is calculated as follows:

(lbs BOD_5/lb MLVSS/day)

$$\frac{\text{Food (F)}}{\text{Microorganisms (M)}} = \frac{\text{Weight of } BOD_5 \text{ added to aeration/day}}{\text{Weight of aeration MLVSS}}$$

Maintenance of F/M ratio, by controlling the aeration MLVSS concentration, is an important process control function. As the F/M ratio is lowered from a value of 1 to 0.1 lbs BOD_5/ lb. MLVSS/day, the treatment efficiency of the activated sludge process improves. For carbonaceous (BOD_5) removal, conventional and contact plants should operate at an F/M range of 0.2 to 0.4. If full nitrification is required as well, ratios less than 0.25 must be consistently attained in the process. Values for extended aeration systems are normally stated as less than 0.1. High-rate systems are designed to operate at F/M's higher than 0.6 and as a result offer only marginal treatment.

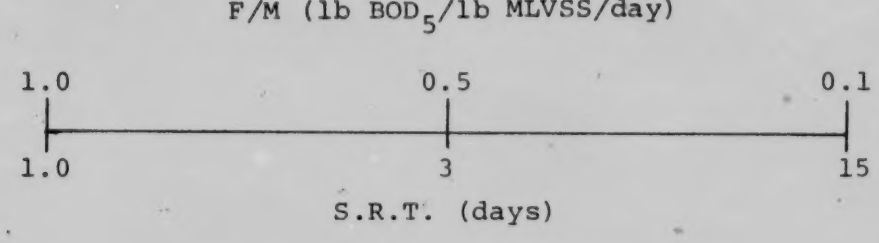
When operating an activated sludge plant, it is imperative that the particular process be maintained within the recommended F/M ranges; otherwise, the aeration mixed liquor flocculation may be disrupted or filamentous organisms may develop causing poor sedimentation. Consequently, a schedule of continuous sludge wasting at the amounts required to maintain the aeration MLVSS in balance with the expected influent BOD_5 concentration is important.

Solids Retention Time (S.R.T.)

The length of time that biological solids are held within the process is known as the Solids Retention Time (S.R.T.) or Sludge Age. This factor is stated in days and is calculated as follows:

$$\text{S.R.T. (days)} = \frac{\text{Weight of aeration MLSS}}{\text{Weight of wasted + Effl. MLSS/day}}$$

Just as with the F/M ratio, S.R.T. can be used to design or maintain an activated process for a given treatment proficiency. As the S.R.T. is raised and F/M is decreased an improvement in wastewater treatment will result.



For BOD_5 removal, conventional and contact plants should be designed and operated in a S.R.T. range of 2 to 5 days with the upper limit used for winter operation. If nitrification is also required year-round then process S.R.T.'s over 5 day must be utilized. The high-rate and extended aeration process appear at the opposite ends of the S.R.T. range with the high-rate less than 2 days and the extended at more than 15 days.

Aeration Dissolved Oxygen (D.O.)

To support the biological oxidation reactions in the activated sludge process, sufficient dissolved oxygen (D.O.) concentration must be maintained in all areas of the aeration basin. A minimum aeration D.O. value of 1 mg/l is usually sufficient to achieve carbonaceous (BOD_5) oxidation, but 2 or 3 mg/l as an operational safety factor is recommended due to daily fluctuations in wastewater loading. To achieve a high level of nitrification, if required, a minimum aeration D.O. of 2 mg/l is required. DO greater than 2.0 mg/l in the activated sludge will not harm the process, but may increase operating costs.

As the volumetric loading of the activated sludge system increases, the need for oxygen (oxygen utilization) to sustain bacteria in the aeration basin rises. Generally, a stable aeration mixed liquor will produce an oxygen utilization rate or respiration rate in mg O_2/l/hr comparable to the volumetric process loading in lbs BOD_5/1,000 cu.ft./day. e.g. 20 lbs BOD_5/1,000 cu.ft./day should produce approximately a 20 mg O_2/l/hr utilization rate in the aeration mixed liquor.

DO Application

Atmospheric oxygen is transferred to the wastewater to aid in the biological oxidation of waste and to prevent the aeration (aerobic) process from going into an anaerobic state, creating septic conditions. This transfer can be accomplished either by pumping air through water as in diffused air systems, or by pumping the water through the air as with surface (mechanical) aeration systems. In either case, the rate of oxygenation is directly related to the rate of pumping.

All of the oxygen in the air is NOT dissolved in the mixed liquor by any of these methods. Only *3 to 10 per cent* of the oxygen is put into solution. This is the reason why such large quantities of air are used to provide sufficient oxygen to maintain an aeration system.

Methods Used to Add Oxygen

1. Diffused-Aeration

   These units (See Figures 2-1 and 2-2) release air bubbles through a porous medium, orifices or hydraulic shear devices such as a sparger or diffuser. The porous media may be tubes or plates constructed of carborundum or tubes tightly wrapped with a plastic such as nylon or saran. The tubes are placed near the side wall of the aeration tank and generate a rolling motion of wastewater to ensure thorough mixing, and they receive air from turbines or compressors.

Figure 2-1

Mechanical Surface Aerators in Operation

Figure 2-2

Close-up of Surface Aerator in Operation Showing Typical Splash Pattern

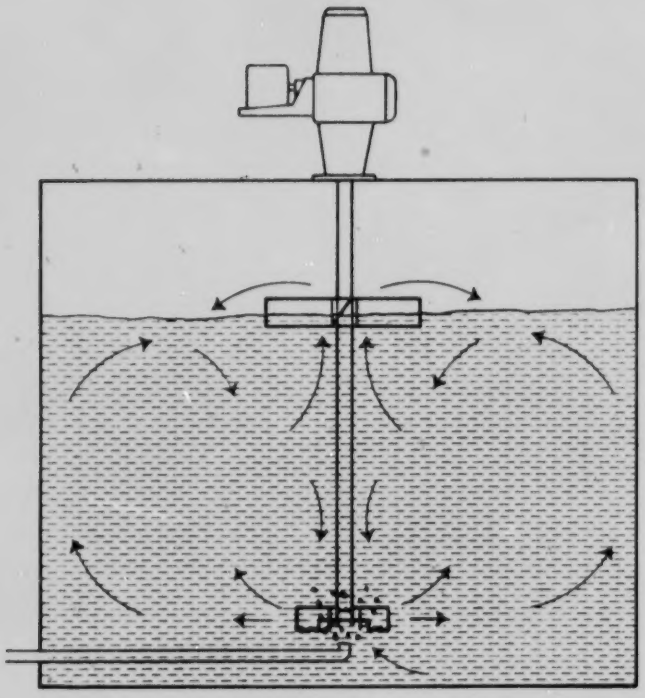
2. Turbine Aerators (See Figure 2-3)
These disperse compressed air by the shearing and pumping action of a rotating impeller. Air is usually fed to the turbine through a sparger ring located underneath the impeller blades.

3. Surface-Aeration
These designs (See Figures 2-4 and 2-5) use rotating blades near the surface of the water to dissolve oxygen from the atmosphere. Air is sucked in at the vortex of the impeller and large volumes of liquid sprayed over the surface of the tank. Some designs employ a draft tube; others use only a surface impeller. Rate of oxygen transfer varies with the impeller diameter, speed of rotation and the depth of blade submergency, i.e. rate of pumping.

Sludge Return Rate

The rate at which settle mixed liquor (sludge) is returned to the aeration basin from the secondary clarifier will effect somewhat the performance of an activated sludge system. The return rate is usually stated as a percentage of the incoming waste flow and in the past 25% was felt adequate. More recent studies have shown that sludge return rates of 80% or more help to maintain more consistent levels of suspended solids in the aeration basin thus maintaining more stable process conditions.

If nitrification takes place in the activated sludge process, the nitrates formed by nitrogenous oxidation may break down into nitrogen gas (denitrification) in the secondary clarifier. Denitrification is more apt to develop during the summer when wastewater temperatures are high (above 15°C) and the clarifier sludge blanket becomes anoxic (low in oxygen). Sludge blanket denitrification causes a gassy scum formation on the surface of the clarifier, but this occurrence can be counteracted by keeping the sludge return rate above 80%. This procedure facilitates the removal of the clarifier sludge before anoxic conditions set in.



Turbine Aerator
Sparger ring below
impeller blades

Turbine Aerator with draft tube for use in tanks over 17 to 20 feet deep.

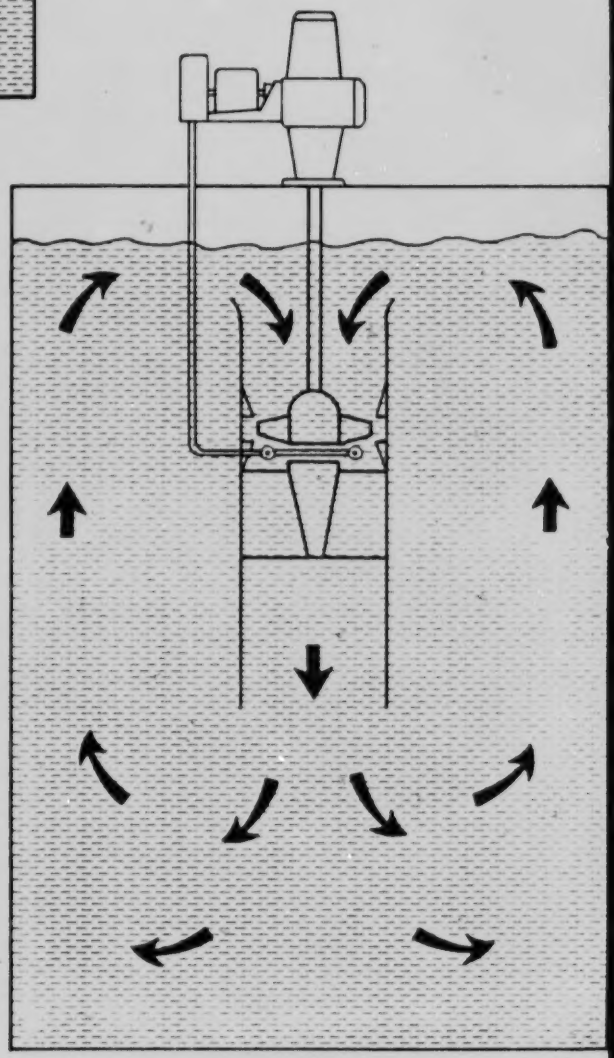


Figure 2-3 TURBINE AERATORS

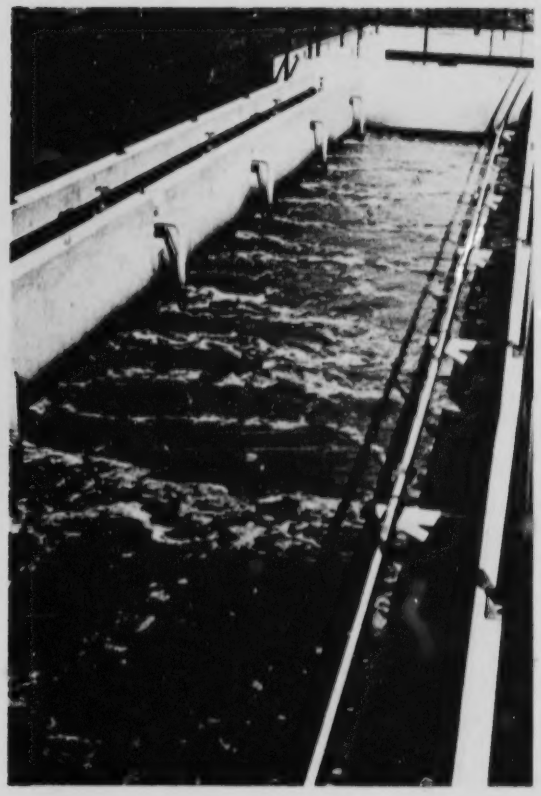


Figure 2-4

Diffused Aerator
System in Operation

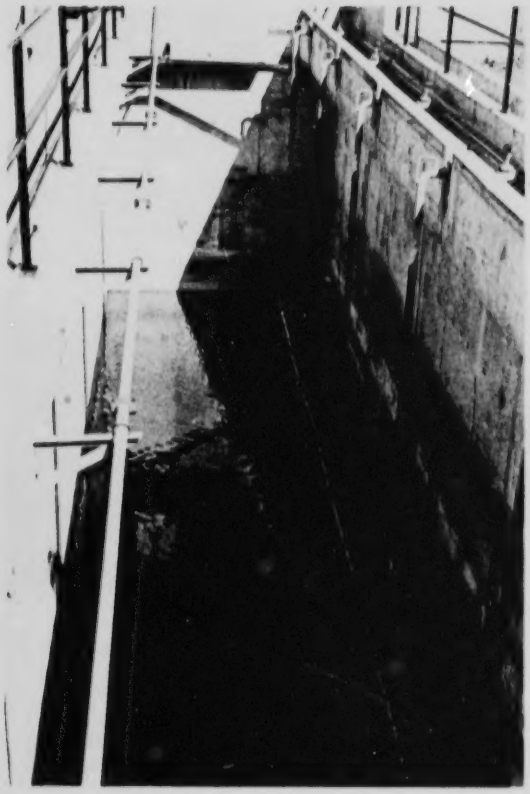


Figure 2-5

Empty Tank Showing
Aeration Devices

Temperature

Wastewater temperatures imposed upon the activated sludge process can have some effect on sedimentation and biological activity. It is generally known that as temperature decreases liquid viscosity increases; consequently, some adverse effect upon secondary clarifier sedimentation may be experienced during winter months.

Bacteria multiply much slower as the sewage temperatures fall below 15°C; therefore, longer detention times are required in the aeration basin to maintain sufficient numbers of microorganisms. Usually an aeration hydraulic detention of 8 hours is sufficient to maintain biological treatment efficiency at low temperatures. Also, at lower liquid temperatures oxygen transfer in the aeration basin is improved and mixed liquor D.O.'s are easier to maintain.

Wastewater temperatures exceeding 20°C during summer months can also cause problems, particularly in an organically overloaded plant. Oxygen transfer is decreased with raised temperatures and this factor, plus enhanced bacterial activity, causes increased oxygen utilization. Consequently, aeration basin dissolved oxygen levels may frequently fall below 1 mg/l and waste treatment will suffer.

Generally, the range of wastewater temperatures experienced in Ontario will not affect BOD_5 (carbonaceous) removal if the waste treatment facility is adequately designed and maintained. But process recovery following a bacterial upset will be delayed at low temperatures and effluent disinfection will be deterred.

If a high degree of nitrogenous oxidation (nitrification) is required, an increase in process S.R.T. is required during winter months to compensate for low temperatures.

Aeration pH

Biological oxidation reactions in the aeration basin, because of their dependence on microorganism quantity and activity, will proceed more efficiently when the mixed liquor pH range is held within 6.5 to 8.5. Aeration pH above or below these values will cause death to a number of microorganisms and result in a reduced process efficiency.

Industrial Wastes

Certain industrial wastes are toxic to aerobic microorganisms and therefore can severely inhibit the performance of the activated sludge process. These wastes can be generally catagorized as being either heavy metal or organic compounds.

Heavy metals such as lead, copper, nickel and chrome are more toxic to microorganisms in the soluble state above 1 mg/l at pH's below 7.0, although recent studies have shown copper to be also toxic in the insoluble state. Organic compounds such as phenol, ether and cyanides are extremely toxic to microorganisms and their concentrations applied to the aeration basin must be limited (See Appendix A for further information.)

High strength wastes (anything greater than 500 mg/l of BOD_5) such as canning or milk products can cause a marked increase in process loading. Resultant process F/M higher than design ranges and oxygen depletion in the aeration basin can occur. Under these conditions a deterioration in floc formation with filamentous organism growth will occur, and aeration mixed liquor sludge volume index (SVI) will likely rise above the optimum range. (Refer to Topic 4)

Summary

Table 2-2 summarizes and compares the characteristics of the three principal types of activated sludge processes.

Table 2-2 COMPARISON OF PLANT TYPES

PARAMETERS	CONVENTIONAL	CONTACT	EXTENDED
Aeration Detention	6-8 hrs.	3-8 hrs.	18-24 hrs.
Volumetric Loading	30-50 lbs/ 1000 cu.ft.	30-50 lbs/ 1000 cu.ft.	5-15 lbs/ 1000 cu.ft.
F/M	.2-.4	.2-.4	≤.1
S.R.T. (BOD_5 Removal)	2-5 days	2-5 days	>15 days
S.R.T. (Nitrification)	>5 days	>5 days	>15 days
S.R. Rate	>80%	25% >100%	>80%
Aeration D.O.(BOD_5)	>1 mg/l	>1 mg/l	>1 mg/l
Nitrification	>2 mg/l	>2 mg/l	>2 mg/l
Primary Sedimentation	✓	NO	NO
Grit Removal	✓	✓	✓
Sludge Wasting	✓	✓	✓
Sludge Reaeration	NO	✓	NO
Sludge Recirculation	✓	✓	✓
Mechanical Aeration	✓	✓	✓
Diffused Aeration	✓	✓	✓

GENERAL CHARACTERISTICS AND PROBLEMS OF INDUSTRIAL WASTES

INTRODUCTION

Contaminants in a waste may be divided into two general groups:

1. contaminants in suspension; and
2. contaminants in solution.

Treatment plants normally remove suspended materials first and then handle the dissolved components. The characteristics of industrial wastes will be divided into categories such as solids, metals, cyanides, greases, oils, and colour.

SOLIDS

Solids in a waste can be subdivided into suspended solids and dissolved solids. Suspended solids consist of particulate matter entrained in the waste and varying in size from small particles, such as measured in the micron range, to larger particles, such as wood fibre from the pulp and paper industry, fruit and vegetable particles from the canning industry, and salt and sand from gravel washing operations. Dissolved solids consist of all materials held in solution.

Laboratory tests are used to determine the amount of solids contained in any wastes. A portion of the waste (aliquot) is taken, using a sample bottle. Following standard procedures, the waste sample is filtered under controlled conditions. The particulate matter caught and left on the filter paper is the suspended solid. The liquid which passed through the filter paper is evaporated. The material remaining in the crucible after evaporation is the dissolved solid. The sum of the suspended solids and dissolved solids is the total solids contained in the waste. All concentrations are recorded in milligrams per litre (mg/l) or parts per million (ppm).

Suspended Solids

Suspended solids can be divided into settleable solids and non-settleable solids. Little difference will be made here, since the effects of settleable and non-settleable solids are almost the same. Settleable solids and the rate of settling are important when designing treatment systems that remove solids by sedimentation. The size of the treatment facilities depends on the settling rates of the entrained matter. The amount of settleability depends upon quiescence, temperature, density and other factors.

Effects of Excessive Suspended Solids on Watercourses

Wastes containing excessive amounts of suspended solids discharged to the watercourse can kill fish by simple abrasive action and by clogging gills and other vital respiratory passages. Suspended solids can destroy benthal organisms and roe, and can ruin spawning beds if they blanket a stream or lake bottom. A heavy discharge of suspended solids can cause turbidity, preventing the passage of sunlight necessary for healthy growth of vegetation in the watercourse. Suspended solids removal from a waste discharge is therefore essential.

Effects of Suspended Solids on Sewage Treatment Plants

Suspended solids can cause serious problems at sewage treatment plants. Abrasive materials can wear out sewers, piping, and pumps, causing extensive maintenance problems. A heavy suspended material that can settle out readily and harden on the bottom of the clarifiers can damage the rake mechanism. Excessive solids in the primary treatment increase the solids handling problems in plant operations.

An excessive amount of readily biodegradable suspended solids could be introduced to a secondary treatment system. Enmeshed with biological floc, it could settle out prematurely in the most quiescent corners of the aeration chamber and begin to turn septic.

A secondary treatment system requires the return of some sludge from the clarifier to the aeration facilities. If this sludge contains a high percentage of inert suspended solids, insufficient active sludge may be recycled. The operation of the secondary stage could be upset. Sludge recycle rates have to be manipulated for an effective treatment plant.

Dissolved Solids

Dissolved solids normally include such waste components as carbonates, bicarbonates, chlorides, sulphates, calcium, magnesium, sodium, and potassium. Generally, these materials are not serious pollutants; however, if their total concentration is high, some problems may result. Dissolved solids can also include dissolved metals and toxic organic compounds. These materials are discussed individually elsewhere because they show specific adverse effects at low concentrations.

Effects of Dissolved Solids on a Watercourse

Dissolved solids can create problems in a watercourse if discharged in excessive concentrations. Some solids can impart a taste or odour to the water and render it unfit for human consumption. High dissolved solids concentrations can alter osmotic pressure and create stresses in microscopic aquatic mechanisms resulting, for instance, in ruptured celled membranes.

Effects of Dissolved Solids on Sewage Treatment Plants

Dissolved solids, except in extreme cases, have little or no effect on proper sewage treatment plant operation. High chloride concentrations, for example, have been discharged to a sewage treatment plant without any apparent adverse effects. If the flow of high concentrations of dissolved solids fluctuates greatly, the result is a shock load effect that could upset the process. These occurrences are usually infrequent.

BIOCHEMICAL OXYGEN DEMAND (BOD)

An oxygen demand in a waste can be exerted by:

- Carbonaceous organic material used as a source of food by aerobic organisms.
- Oxidizable nitrogen derived from nitrate, ammonia, and organic nitrogen compounds which can serve as food for bacteria.
- Certain chemical reducing compounds which normally are associated with industrial waste discharges.

Eventually, these compounds will be oxidized by bacteria, at which time the dissolved oxygen in the water is used up. The amount of oxygen consumed is a measure of the BOD. The longer the material remains in the water, the more oxygen it will consume. The length of time used to measure BOD is normally set at 5 days, hence the more exact term is 5-day BOD (BOD_5).

High BOD demands are significant. They can cause septicity. They can decrease oxygen levels to the point where the water cannot sustain a biological community. In lakes or slow moving streams, septic conditions can lead to turbidity, odour problems, and unsightly conditions. The discharge of BOD material to a watercourse must therefore be controlled.

The BOD parameter is essential in sewage plant design and operation. One of the main objects of the sewage treatment plant is to stabilize the waste. This, in effect, means removing all, or most, of the materials exerting a BOD. The amount of BOD requiring stabilization dictates the size of the aeration basin, sets out the amount of oxygen that must be supplied to the waste, limits feed rate, established sludge recycle rates, etc. By doing BOD analyses at the various process stages at the sewage treatment plant, the operator can evaluate the operation of his plant.

If high quantities of materials are discharged to a sewer exerting a high BOD, they can overtax existing aeration facilities at a sewage treatment plant. Insufficient oxygen levels in the plant will lead to insufficient dissolved oxygen levels in the overflow to the secondary clarifier. The higher BOD loading may affect sludge characteristics and produce a poor settleable sludge. Only partial treatment of wastes will occur and a poor quality effluent is released to the watercourse.

CHEMICAL OXYGEN DEMAND (COD)

The BOD test cannot be used for on-the-spot checks, since it takes 5 days to carry out. The chemical oxygen demand (COD) represents the amount of oxygen required to stabilize a waste chemically. Similar to BOD, the COD measures the effects of a combination of materials and does not reveal the concentration of a specific substance. BOD and COD relationships in a waste should be determined before conducting lab tests. This makes it possible to perform COD analyses, estimate BOD concentrations, and check on the operation of the treatment system. COD tests may exclude some organic compounds in the waste which could exert a BOD (e.g. acetic acid).

METALS

Metals are found in different forms in wastes. They may be found in solution as ions, or in suspension as insoluble hydroxides or insoluble salts. Metals may be present therefore as suspended or dissolved solids. It is best to discuss them separately on an individual basis, however, because they are normally very toxic. Special treatment systems have to be designed to remove them from the waste.

Metals are normally introduced to a watercourse as a result of an industrial waste discharge. In some cases, natural leaching of a metal into a watercourse may occur if there are ore bodies exposed to natural elements such as rain, frost, erosion, etc. As a general rule, metals are present in a sanitary sewer as a result of an industrial waste discharge.

Metals may be extremely toxic both to biological life in a watercourse and to bacterial sludge necessary in sewage treatment plant operation. Because of their toxicity, metals have been responsible for numerous fish kills in the Province. Metals also exhibit synergistic effects; that is, one metal can increase the toxicity of another so that their combined concentration is more lethal than if either metal were found in a watercourse by itself.

Metals can also disrupt the proper operation of a sewage treatment plant. In extreme cases, bacteria in a biological system may be killed entirely. At lower concentration levels, metals may be picked up by bacterial sludge and may accumulate in the digester to toxic levels at which the digester could no longer function.

Some of the more common metals found in wastes are listed in Table 2-3.

ORGANIC COMPOUNDS

A number of organic compounds in solution require special attention because they exhibit their own peculiar traits. Some are very toxic, such as phenol and cyanide. Oils and greases can also be harmful.

PHENOL

Phenol is a colourless crystalline substance with a characteristic medicinal odour. It is used as a disinfectant and in the manufacture of various plastic resins. It is very soluble in water. As a result, it can become a serious contaminant.

Phenol in domestic waters can cause a serious nuisance problem. During the chlorination of drinking water, phenols also become chlorinated. Even at extremely low concentrations (0.002 mg/l) phenols produce an objectionable taste. This taste has upset many operations at breweries, distilleries, and soft drink bottling plants.

Table 2-3

SOME COMMON METALS FOUND IN INDUSTRIAL WASTES

METAL	EFFECT ON WATERCOURSE	EFFECT ON SEWAGE TREATMENT PLANT	COMMENTS
Chromium	Toxic to fish in concentrations from 5 ppm to 10 ppm	Toxic to biological sludge in concentrations in excess of 10 ppm	Found in hexavalent and trivalent state
Copper	Toxic range to fish is 0.1 ppm to 1 ppm	Toxic to biological sludge	Exhibits synergistic effects with zinc
Iron	Toxic range to fish is 0.2 ppm to 2 ppm	Less toxic to biological sludge	Easily forms hydroxide and settles out in primary treatment works
Lead	Toxic range to fish is 0.1 ppm to 5 ppm	Toxic to bacterial sludge	Metal can accumulate in fish tissue
Nickel	Toxic to fish	May be toxic to bacteria in 2 ppm	Forms complex with cyanide
Zinc	Toxic range to fish is 0.1 ppm to 1.0 ppm	Toxic to bacterial sludge	Exhibits synergistic effects with copper

Phenols in a watercourse are toxic. They can cause extensive fish kills even at concentrations in the 0.1 mg/l range. At lower concentrations, they are toxic to lower biological forms, thus destroying the delicate balance of an aquatic environment.

Phenols at high concentrations can cause a shock load and be toxic to bacteria used in activated sludge treatment plants. After being properly acclimatized, a secondary sewage treatment plant can be used to treat phenolic discharges. The feed rate to the plant should be maintained at near constant levels to eliminate shock load and withdrawal effects. Biological systems have successfully treated industrial wastes containing high phenol concentration.

CYANIDE

Cyanide is introduced to the waste in various forms. All of these forms liberate the toxic hydrogen cyanide gas, characterized by an almond smell, whenever the pH of the effluent drops below a value of 8. Cyanide is used extensively in plating industries. It is normally associated with other metals such as copper, zinc, and cadmium. Cyanides may also be discharged from gas works, coke ovens, and chemical industries.

Cyanide is extremely toxic to fish and has been responsible for numerous fish kills. Cyanide can increase the toxicity of metals and hence render normal background levels of metals lethal to aquatic life.

The discharge of a strong cyanide waste to a sanitary sewer is a potential health hazard. Cyanide gas can be liberated from the waste; it can become trapped in poorly vented manholes and other dead areas in the sewers and become a real hazard to sewer maintenance workers. Cyanide is extremely toxic to bacteria. It can create operational problems at the sewage treatment plant. Enough cyanide in the waste can kill off the bacteria in the secondary stage, necessitating a new start up of operation. The introduction

of small amounts of cyanide into a treatment plant at constant levels may allow the bacteria to become acclimatized and treat the waste quite effectively.

OILS AND GREASES

Oils and greases in wastes are commonly denoted by the term ether solubles or hexane solubles. The term does not differentiate between materials of animal or mineral origin. Since oils and greases act very similarly, they can be readily discussed and denoted by the same term. Oils and greases having an animal or vegetable origin are somewhat less noxious since they are more biodegradable.

Large quantities of oil discharged into a watercourse can create extensive damage. Beaches covered with oil become unsightly and cannot be used for recreational purposes. Water fowl, weeds, and other vegetation covered with oil cannot continue their normal life functions and die. Small quantities of oil can spread and cover large surface areas of water. This prevents the proper penetration of light so necessary for proper weed growth. In domestic water supplies, oils can impart unpleasant taste and odours rendering the water unfit for human consumption. Treatment is necessary to prevent the discharge of these materials to a watercourse.

Large quantities of grease and oil can create problems in a sewage plant. Grease can combine with other solids and produce large round solid masses more commonly known as "grease balls". A sufficient number of these can plug sewers and lines creating maintenance problems. Smaller grease balls floating on the surface of clarifiers and aerators create an unsightly mess. Their removal becomes an extra procedure in plant operation.

Oils also form unsightly scums on surfaces of clarifiers and aerators. Oil can cover equipment, plug diffuser air nozzles and create other maintenance problems. In sewage treatment lagoons, oil can cover the surface of the pond and interfere with the oxygen transferring mechanisms.

Extensive quantities of oil become a fire hazard, especially if the oil contains gasoline that can evaporate and form an explosive mixture with air.

Steps should be taken at the source to prevent excessive oil discharges to the sanitary sewer.

NUTRIENTS

The term nutrient normally refers to nitrogen and phosphorus compounds in the waste that promote the growth of weeds and algae. An overabundant growth of weeds and algae creates nuisance problems in recreational areas. Masses of algae rotting on shore create odour problems, destroy the value of the water-front property and make the area uninhabitable. Treatment systems have now been designed to remove these materials from the waste.

COLOUR AND TURBIDITY

Colour and turbidity are useful conditions when determining the nuisance values of some wastes. Strongly coloured effluent, for example, from a dyeing industry, may impart an objectionable colour to a lake or river and destroy its aesthetic value. Highly turbid streams can prevent a proper passage of light necessary for good aquatic growth. In some cases, dyes can be broken down quite readily in a sewage treatment plant. In most cases, however, the more modern dyes are extremely stable and can only be handled by dilution.

Determining Toxic Levels

In some cases, it is difficult to assess the nature of a particular waste and predict its harmful effects on aquatic life. A toxicity test is carried out to determine whether such a waste can be released to a watercourse. This test involves immersing minnows into various dilutions of the waste and studying the effects on the fish. If the fish survive, the effluent is considered non-toxic. If the fish die, the dilution is noted and used to set an effluent quality standard. With more and more new chemicals being created, the toxicity test has become a handy tool to evaluate some of the more exotic types of wastes.

OBJECTIVES:

The trainee will be able to

1. Recall the purpose of sampling and flow measurement.
2. Define and recall the purpose of
   a) grab sample
   b) composite sample.
3. Recall the location and type of samples to be taken in a plant.
4. Define
   a) Open channel flow conditions
   b) Full pipe flow conditions.
5. Recall the components comprising the system used in open channel flow measurement and the purpose of each.
6. Perform the graphical integration procedure to determine total flow.
7. Discuss the relationship of flow measurement instrumentation size to error in recorded flow rates.
8. Describe the method for the calibration check of flowmeters for open channel flow measurement.
9. Recall the purpose and basis of the operation of a magnetic flowmeter.

SAMPLING AND FLOW MEASUREMENT

PURPOSE

Sewage is treated to produce an effluent which will not impair the quality of the environment. If spending money is necessary to reduce pollution, then a knowledge of plant performance is necessary to justify the cost. *The purpose of sampling and flow measurement is to obtain data concerning the physical, chemical and biological characteristics of the waste stream. This information can be used*

1. *to determine plant loadings,*
2. *for control of the individual processes,*
3. *to demonstrate compliance with regulations or standards,*
4. *to estimate the effect of plant effluent on the receiving waters,*
5. *for the design of plant extensions.*

SAMPLING

TYPES OF SAMPLES

Grab Sample

A *grab sample* can be defined as a single sample of wastewater taken without considering the time or the rate of flow. It is not very useful for calculating a waste loading since a single aliquot is not usually representative of average conditions. *Grab samples are only of value in determining the composition of either maximum or minimum flows.*

Composite Sample

A *composite sample* is defined as one which is built up, or composited from a series of grab samples taken at intervals during a fixed sampling period. It represents the average characteristics of the waste flow over the survey period and may be used to calculate waste loadings using the flow volume over the same period.

Composite samples are of the two types:

1. Those in which the grab samples are kept separate and analyzed separately to enable variations to be determined.

2. Those in which the grab samples are combined to form a bulk sample, all or part of which is subjected to analysis.

In the first type, a constant volume is taken at each interval regardless of the flow rate at that time. However, if a waste loading is to be calculated and it is known that the flow rate is not constant, the flow rate must be measured when each sample is taken.

In the second type of composite, where the aliquots are combined, the amount of the aliquot taken at each interval must change in proportion to the flow rate *at the time when the sample is withdrawn from the waste stream.* For example, if at one sampling time the measured flow rate is 150,000 gpd and an aliquot of 500 ml is taken, at the next sampling only 250 ml should be taken if the measured flow rate is 75,000 gpd. *A constant volume is taken at each interval only when the flow rate is constant.*

A constant time interval is usually chosen between aliquots of a composite sample, since most variations in waste characteristics occur on a time cycle. The time allowed between aliquots is determined by the variability of waste characteristics. If the characteristics vary rapidly, the aliquots must be taken frequently, say every half hour, while if the waste is of fairly uniform character, the intervals may be longer, say every hour. The most accurate average will, of course, be given by a continuously drawn sample, but unless automatic equipment is used, this will not be practical. Therefore, longer intervals, such as 10, 15, or 30 minutes are often used. However, aliquots should never be taken less than once per hour. A similar principle applies when characteristics vary with volume.

To give an accurate picture of the overall quality of raw sewage entering, of plant effluent leaving, and of the processes going on within the plant, a series of three 8-hour composite samples over a 24-hour period is the ideal, and is strongly recommended. Daylight hour 8-hour composites are the next best choice, if sampling cannot be done regularly at night. In this case the occasional night composite should be attempted, even if this requires shortening a day shift to accommodate the extra hours worked.

TYPES OF SAMPLING DEVICES

There are two types of sampling devices, *automatic* and *manual*. Examples of each with their advantages and limitations are as follows:

1. Automatic

   a) Vacuum Sampler

      This apparatus consists of an evacuated sample container, an electrically (or mechanically) operated closing device and a length of tubing. The open end of the tube is placed in the waste stream and the timing mechanism is set to operate at the required intervals. When the closing device is opened, wastewater is drawn into the container.

      Advantages of this apparatus include simplicity, reasonable cost and ability to function for long periods on a small storage battery. Its chief limitation is that it can only be used on a minimum lift.

   b) Pump Sampler

      This apparatus consists of a sample container, a pump, and interconnecting tubing. Many types of pumps may be used, among the most useful being chemical feed pumps, due to their ability to meter accurately small volumes of liquid. A timing mechanism may be incorporated.

      Both vacuum and pump samplers may be equipped to sample in proportion to the flow rate.

In addition to the two types described above, there are many others based on similar principles and on various types of wheels, discs and rotating scoops.

2. Manual

Equipment used in manual sampling is simple and consists of bottles, lines, poles (able to be joined together), bucket or bottle holders, and weights.

COLLECTION OF SAMPLES

In keeping with the prime objective of a sampling programme (to obtain accurately representative samples), certain precautions must be taken to avoid errors. Whatever type of container is used, it must, of course, be "clean"; therefore rinse with sample to be taken, or clean water.

NOTE: When samples are taken through the plant, a good technique is to start with final effluent, then to the primary effluent, raw sewage, activated sludge, and sludge return, raw sludge in digester samples. This sequence will prevent cross-contamination of samples. Where smaller samples are to be taken from a larger sample, care should be taken that the sample is representative (shake or stir sample well to ensure a thoroughly mixed smaller sample).

SAFETY IN SAMPLING

No sample is worth obtaining at the risk of life and limb. Safety precautions should be observed at all times, including the following:

1. Never sample alone at night where lighting is poor.

2. Never enter a tank or other vessel or a sewer unless it cannot be avoided, and then *only when it is known that the atmosphere is free from noxious gases and there is no possibility of any material entering while sampling is in progress.*

Never do it alone and always use a lifeline.

3. Always remember that most organic liquids are highly inflammable and form explosive mixtures with air. *Smoke only in safe places and when sampling is completed.*

4. All chemical substances must be considered harmful (until proven otherwise) if ingested into the stomach or lungs, or by contact with the skin and eyes. It is vital to know what materials may be encountered during a survey and to use all necessary protective devices.

WHERE TO SAMPLE AND TYPE OF SAMPLE REQUIRED

REMEMBER: THE ANALYSIS IS ONLY AS GOOD AS THE SAMPLE TAKEN!

Taking good, representative samples of the wastewater entering and leaving the treatment plant is extremely important. The type of sample, where it is taken and how it is taken require conscientious care and attention since they will greatly influence the reliability of the data obtained.

The following points must be considered when taking a sample as well as where the sample is taken:

1. Raw Sewage, Primary Effluent, Plant Effluent

   Composite samples should be taken wherever possible when sampling raw sewage, primary effluent, and/or plant effluent. A continuous 24-hour sampling period is desirable, and the sample taken should be proportional to the flow. An automatic sampler should be used.

   Because of the variability in raw sewage, it is important that frequent aliquots be obtained of the raw sewage and primary effluent. The same frequency is not as important when sampling plant effluent.

2. Raw Sludge

The composition of raw sludge can vary widely between pumping cycles, even within a single pumping cycle. To get a representative picture of the sludge, use a sample composed of at least three equal-sized grab samples. Take one grab sample at the beginning of one pumping cycle, another grab sample near the middle of a second pumping cycle, and a third grab sample near the end of a third pumping cycle.

3. Anaerobic Digested Sludge - Primary Digestion

The primary anaerobic digester is usually heated and the contents well mixed and nearly homogeneous. A grab sample should be enough to determine the quality of the sludge, and the sample can be taken at some convenient location, *if it is representative.*

4. Anaerobic Digested Sludge - Secondary Digestion

A sample from the secondary digester can be taken when the sludge is removed from tank for disposal or haulage. If possible, a composite sample should be taken, made up of one grab sample at the beginning of the day's pumping, another in the middle, and the third sample taken near the end of the pumping.

5. Digester Supernatant

A sample is taken whenever the supernatant is withdrawn. A composite sample of at least three equal-sized aliquots should be taken: at the beginning, the middle and near the end of the recycle.

6. Aeration Tank (Mixed Liquor)

The aeration tank sample is taken at the effluent end of the aeration system. By necessity, it is a grab sample, taken at about the same time each day to ensure similar hydraulic conditions, (preferably mid-way through peak load conditions).

If there is more than one aeration tank, a sample should be taken from each tank.

For *return sludge*, a grab sample is enough to estimate the sludge solids concentration for wasting purposes.

7. Aerobic Digester

The contents are usually well mixed - a grab sample should do.

FLOW MEASUREMENT

FLOW CONDITIONS

Two general classes of flow conditions exist within a water pollution control plant.

1. Open channel
2. Full pipe or pressurized

Open channel conditions refer to a fluid flowing with a free surface which is at atmospheric pressure. This flow condition is found in plant channels, tanks and in any pipes flowing less than full.

Full pipe or pressurized flow conditions exist when the fluid flowing within a conduit is at greater than atmospheric pressure. This flow condition exists in any pumped lines such as raw sewage, return sludge, raw sludge, etc.

Different flow measurement techniques are used for each flow condition and are described in the following paragraphs.

OPEN CHANNEL FLOW MEASUREMENT SYSTEM

Flow measurement under open channel flow conditions is typically achieved through the *use of weirs or the Parshall flume*. Figure 3-1 indicates the elements comprising an open channel flow measurement system.

WEIRS AND PARSHALL FLUME

The common types of primary flow elements employed for open channel flow measurement are as follows:

1. V notch weir
2. Rectangular weir without end contractions
3. Rectangular weir with end contractions
4. Parshall flume

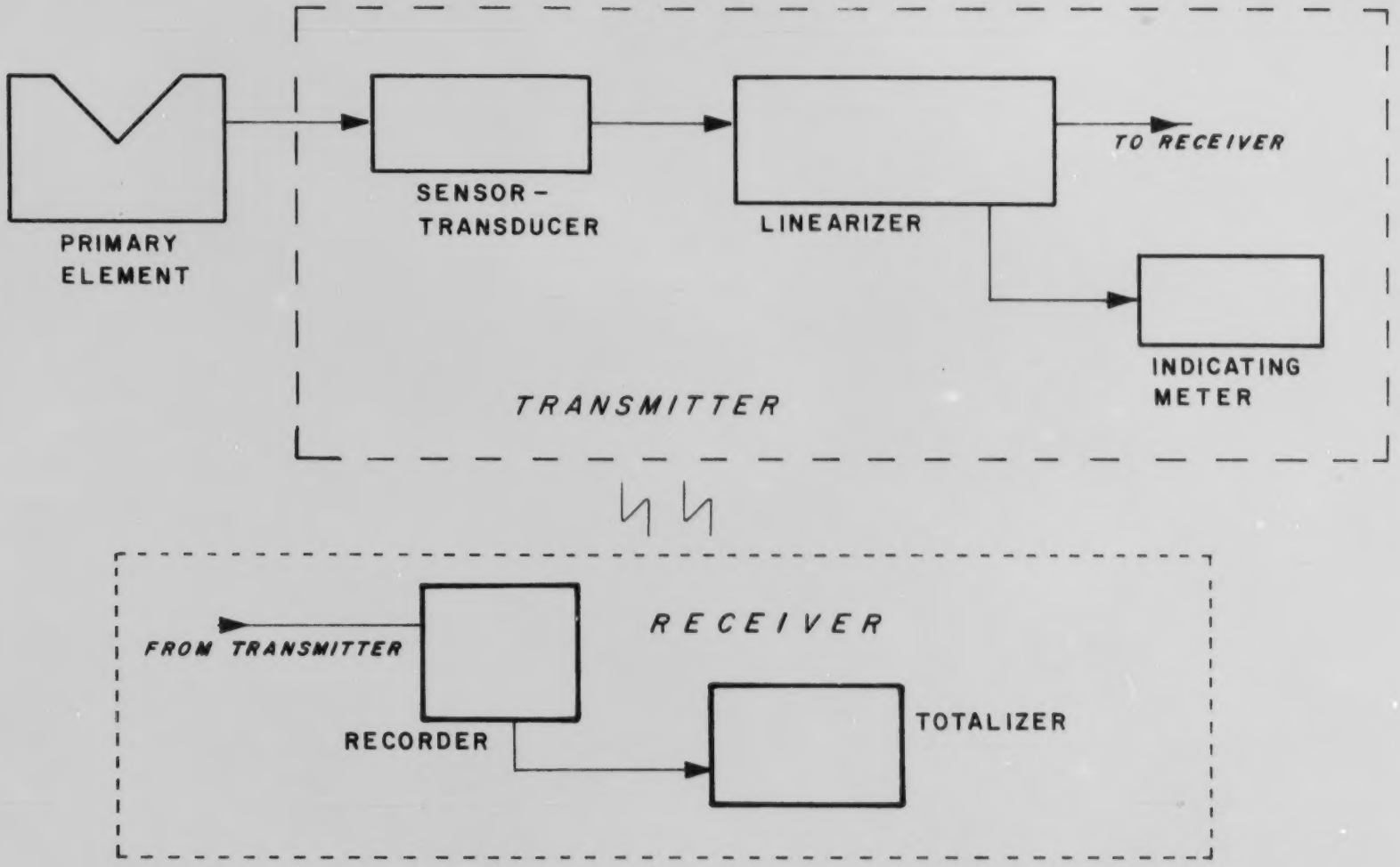
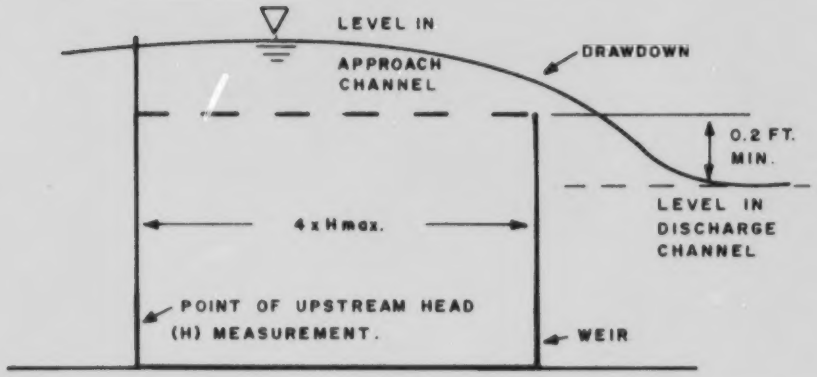
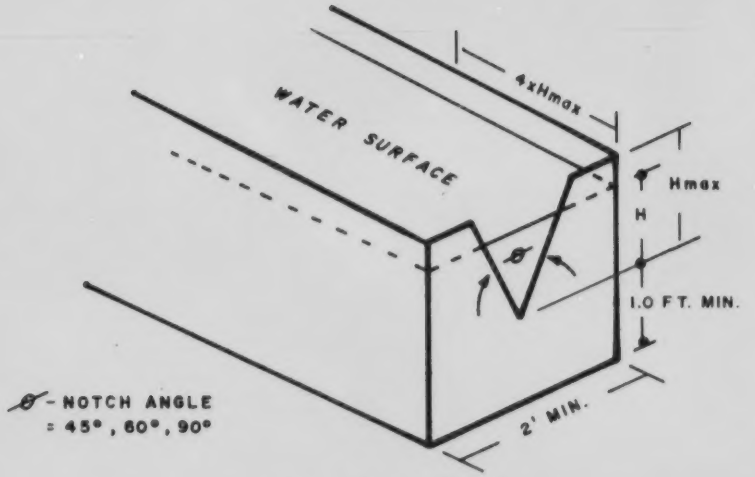


FIG. 3-1 OPEN CHANNEL FLOW MONITORING SYSTEM.

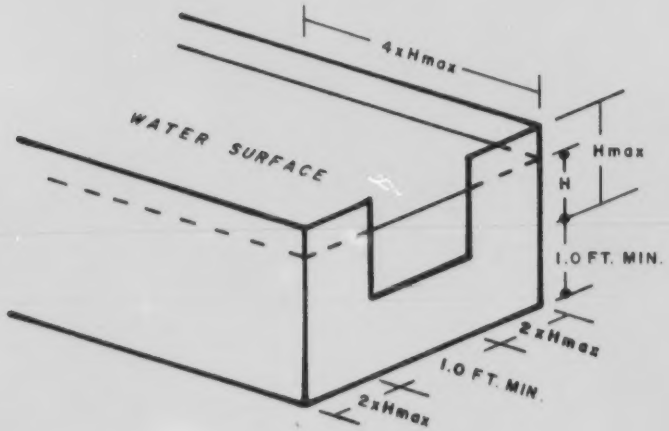


APPROACH CHANNEL IS STRAIGHT, HAS A UNIFORM CROSS-SECTION, AND IS 10xHmax LONG.

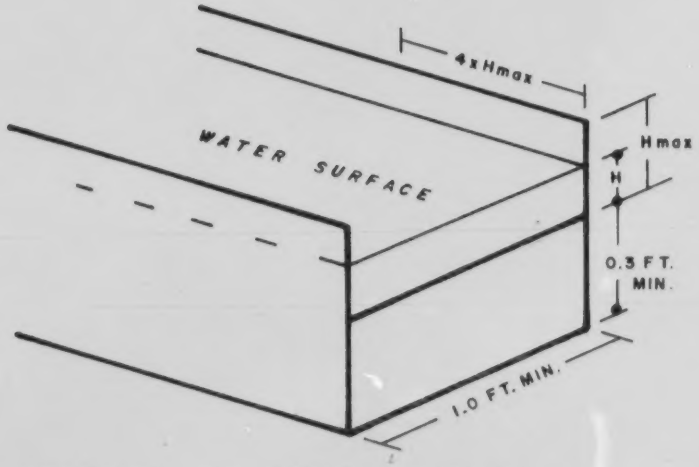
WEIR PROFILE
(APPLIES TO ALL WEIR TYPES)



V-NOTCH WEIR



RECTANGULAR WEIR WITH END CONTRACTIONS



RECTANGULAR WEIR WITHOUT END CONTRACTIONS

FIG. 3-2 WEIRS

The purpose of the primary element is to produce a specific relationship between an upstream depth of flow, (termed the head) and the flow rate. In all cases it is the upstream head that is measured. Flowrate is determined from the specific relationship between head and flow for the particular primary element which is used.

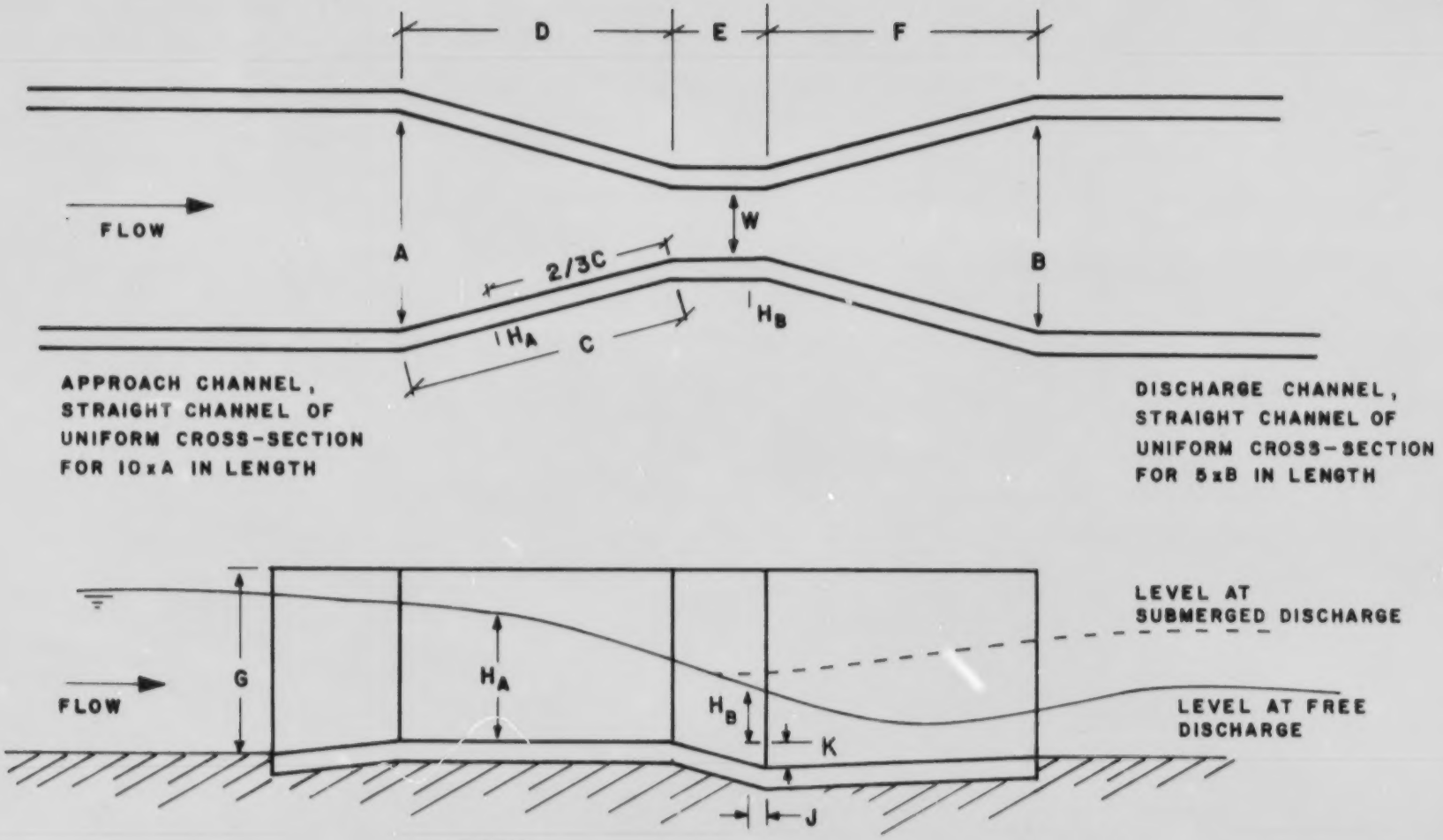
The relationship between head and flow rate for the primary element is in part the result of theoretical calculation and in part the result of years of experimentation and testing. As a result of these efforts, the configuration of the primary elements and the conditions under which they may be employed have become standardized.

In order to accurately determine flowrate from head measurements the following criteria must be met:

1. The configuration of the primary element must be within the limits set by the standard.
2. The configuration of the approach channel must be within the limits set by the standard.
3. The discharge channel must be so constructed as to allow the flow to freely discharge from the device.
4. The range of flowrates (maximum and minimum) for a given size and type of device should fall within the recommended limits.

Figures 3-2 and 3-3 and Table 3-1 indicate the recommended primary element geometry and channel configuration for the commonly employed weirs and the Parshall flume.

By adhering to these standards, an inherent accuracy of 5% is available. This implies that if the head were measured with no error the actual flowrate could be determined to within $\pm$5%.



LEGEND

H_A - POINT OF UPSTREAM DEPTH MEASUREMENT

H_B - POINT OF MEASUREMENT TO TEST FOR FREE DISCHARGE CONDITION

W - THROAT WIDTH WHICH SPECIFIES FLUME SIZE

NOTE: FOR DIMENSIONS SEE TABLE 3-1

FIG 3-3 PARSHALL FLUME

TABLE 3-1 PARSHALL FLUME

DIMENSIONS

W	A	B	C	D	E	F	G	J	K	MIN. FREE FLOW	MAX. CAPACITY
ft. in.	ft. in.	ft. in.	ft. in.	ft. in.	ft. in.	ft. in.	ft. in.	ft. in.	ft.in.	IMGD	IMGD
3"	$10\frac{3}{16}$"	7"	1' $6\frac{3}{8}$"	1' 6"	6"	1' 0"	1' 3"	0' 1"	0' 1½"	.017	.33
6"	1' 3½"	1' 3½"	2' $\frac{7}{16}$"	2' 0"	1' 0"	2' 0"	1' 6"	0' 2"	0' 3"	.025	1.6
9"	1' $10\frac{3}{8}$"	1' 3"	2' $10\frac{5}{8}$"	2' 10"	1' 0	1' 6"	2' 0"	0' 2"	0' 3"	.05	2.8
1' 0"	2' 9¼"	2' 0"	4' 6"	4' $4\frac{7}{8}$"	2' 0"	3' 0"	3' 0"	0' 2"	0' 3"	.22	8.3
1' 6"	3' $4\frac{3}{8}$"	2' 6"	4' 9"	4' $7\frac{7}{8}$"	2' 0"	3' 0"	3' 0"	0' 2"	0' 3"	.27	12.5
2' 0"	3' 11½"	3' 0"	5' 0"	4' $10\frac{7}{8}$"	2' 0"	3' 0"	3' 0"	0' 2"	0' 3"	.38	17.5
2' 6"	4' $6\frac{3}{4}$"	3' 6"	5' 4¼"	5' 3"	2' 0"	3' 0"	3' 0"	0' 2"	0' 3"	.43	21.7
3' 0"	5' $1\frac{7}{8}$"	4' 0"	5' 6"	5' $4\frac{3}{4}$"	2' 0"	3' 0"	3' 0"	0' 2"	0' 3"	.54	26.7

Free flow conditions exist for the following ratios of H_B/Ha

Size	H_B/Ha
3"	< 0.5
6-9"	< 0.6
larger than 9"	< 0.7

Deviation from the recommended standards reduces the inherent accuracy and if the deviation is sufficiently large the flow data can become meaningless.

Sensor-Transducer

The purpose of the sensor-transducer is to detect the variations in the upstream head and translate these variations into either an electrical signal or mechanical movement which is used to generate an input signal, proportional to sewage depth, to the linearizer.

The commonly employed types of sensor are the bubbler and the float.

The bubbler sends a constant stream of air through a tube, called the dip tube, located at the point of measurement appropriate to the primary element. As the liquid depth increases, the back pressure upon the air stream is increased. The response in back pressure is proportional to depth. The back pressure is then translated into a mechanical movement or an electrical signal by means of a bellows or pressure transducer.

Float operated instruments employ either a scow float, which can be placed directly in a flowing stream channel, or a ball type float, which requires a stilling well.

The float rides the surface of the liquid and moves vertically in proportion to liquid depth. In most cases, a counter weighted chain or tape attached to the float produces movement of a pulley which is translated by gears or a potentiometer into either a mechanical or electrical signal.

For bubblers, the dip tube should be rodded out at regular intervals. This is to prevent the buildup of and/or to remove any foreign matter such as algae, grease or rags. These may partially plug the tube increasing air back pressure and producing erroneous readings.

For scow type floats, a simple visual check to ensure that rags or other debris have not been trapped on the float is sufficient to ensure proper float operation. For floats provided with stilling wells, the pipe connecting the well to the channel should be checked by backflushing to ensure that free flow is possible. The stilling well itself should also be checked for accumulated sediment. In the event that sediment interferes with float operation, the well should be removed. It should be noted that most stilling well facilities are provided with a constant stream of purge water, which reduces the likelihood of sensor line blockage and sediment in the well.

Linearizer

The linearizer accepts the level (input) signal produced by the sensor-transducer combination and mechanically or electrically converts the level signal into a signal proportional to the flow rate.

For each primary element there exists a different characteristic relationship between upstream depth of flow and flow rate. It is the function of the linearizer to reproduce this characteristic relationship.

Figure 3-4 illustrates the principle of operation of the linearizer. Figures 3-5 to 3-8, pages 3-22 to 3-25, give the characteristic relationships between head and flow rates for the common sizes of primary elements.

Recorder-Totalizer

In most plants the actual flow measurement takes place at a location remote from the control building. As it is desirable to observe plant flow rates at a central location, a transmitter-receiver combination is employed.

The transmitter consists of the sensor-transducer, linearizer, and some device to transmit the signal to the receiver. The receiver accepts the signal and produces a

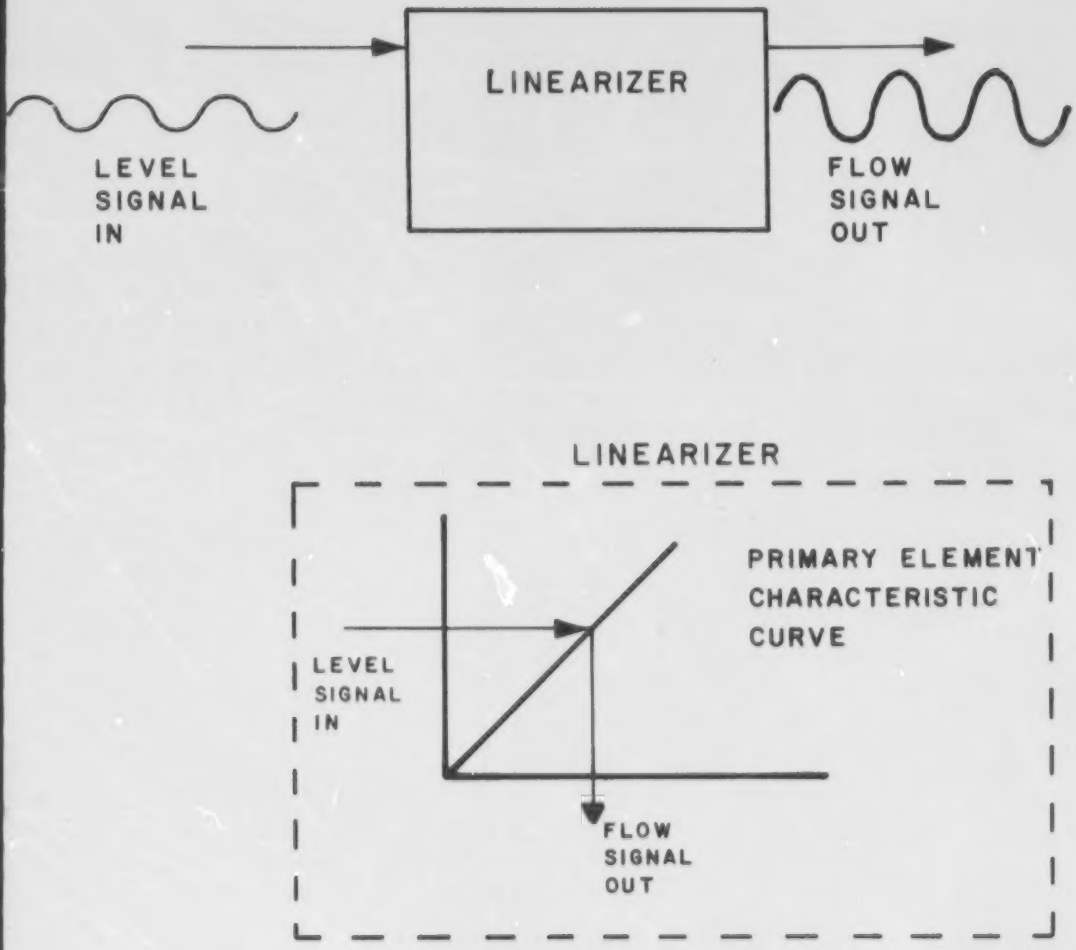


FIG. 3-4 LINEARIZER OPERATION

visual display of the flow rate by means of either an indicating meter or a recorder, or both. In addition, the linearizer output signal is transmitted to the totalizer, which is a counter that indicates accumulated total flow.

The major limitation on the accuracy of the totalizer occurs in rapidly cycling flow, where the counter cannot respond sufficiently rapidly to the flow variation and produces erroneous totals. In this case, the total flow should be obtained from the flow chart by graphical integration and the totalizer is used as only a rough check on the values calculated. The *graphical integration* procedure is as follows:

1. For a given period of time as represented by the totalizer reading i.e. 8 a.m. to 8 a.m. take the appropriate flow chart for the same period.
2. Divide the chart into 1 hour time periods. For each time period determine the average flow rate by inspection.
3. Calculate the volume of flow for each 1 hour period and add all the volumes for the time period of interest.
4. The volume of flow resultant from the *graphical integration* and the totalizer should be compared and any deviation noted.

Additional error may be introduced into totalizer readings from an improperly sized primary element or flowmeter. The error would be of the same magnitude as that associated with the flowmeter.

Instrumentation Size vs Error

The sizing of the instrumentation for flow measurement is another consideration affecting the inherent accuracy of the overall measurement system. Typical accuracies for this instrumentation range up to 5% of the full scale value.

In a properly sized system the average daily flow rate should produce an approximately mid-scale response on the instrument. This ensures that the absolute error of the instrument is minimized and that adequate flow range is available to accommodate maximum and minimum flow rates.

As an example of the possible error introduced by a poorly sized system consider the following case. A flowmeter, with a range of 0 to 10 MIGD and an accuracy of 5%, is used to indicate a 2 MIGD flow rate. The absolute error of the instrument is 5% of 10 MIGD (or 0.5 MIGD). When applied to a reading of 2 MIGD the error is 25%.

When conducting a calibration check of flow measuring equipment the operator must be aware of the possible absolute error introduced by poorly sized flow measurement instrumentation and must record the magnitude of this error.

CALIBRATION CHECK

Flow measurement instrumentation should be serviced by a competent serviceman every year. To ensure that the flow rate data presently recorded is meaningful an operator should attempt to check the overall functioning of the flow metering system at least every three months.

A record of the flow metering checks should be maintained and the observed accuracy noted.

The following presents a check list approach for the examination of flowmeters.

1. Determine whether the primary element is properly installed by means of the data given in previous paragraphs.
2. Determine the absolute error associated with the instrumentation at the maximum, minimum and average flow rates. Use the method given in previous paragraphs.

   Record the magnitude of these errors.

3. Check old flow charts for typical maximum and minimum flows and compare these with the recommended maximum and minimum flows for the particular primary element. If the maximum or minimum flows fall outside the recommended limits, portions of the chart where these flows occurred should be considered suspect.

4. Check the sensor location to establish that it is installed in the correct position, within the channel. In addition, a check should be made to establish whether the sensor is fully operating and not impeded in any fashion by grit, debris, etc.

5. A check of flowmeter calibration accuracy can now be made. In order to perform a calibration check the following equipment is required:

   a) A ruler or measuring stick.

   b) The curve relating head to flow rate for the particular size and type of primary element.

   The procedure is as follows:

   a) Bypass the flow away from the primary element. When all flow has ceased check the reading of the recorder and any indicating meter either at the transmitter or receiver. If the recorder or meter indicates a flow rate other than zero, note the deviation and whether it is positive or negative.

   b) Resume flow to the primary element and allow at least 30 minutes for flow to stabilize. In the case of a Parshall flume measure the depth of flow at the appropriate point and simultaneously have an assistant mark the recorder chart at that point in time. For rapidly cycling flow attempt to make this determination when the flow rate reaches the maximum value and the minimum value.

A number of determinations should be made at the maximum flow rate, minimum flow rate and an intermediate value. For weirs it is usually difficult to measure the head at the upstream gauging point. Accordingly the head is measured at the weir itself and allowance of 1/8" to 1/4" is added to the measured head to compensate for the draw down of the liquid surface at the weir (see Figure 3-2).

c) The head measurements can now be converted to flow rate by employing the appropriate calibration curve. A comparison of the recorded or indicated value and the measured value is made. Any deviation should be noted and if the errors are sufficiently large i.e. greater than 10-15% the instrumentation should be recalibrated by a serviceman.

6. The functioning of the totalizer should also be checked periodically by comparing the total volume indicated by the totalizer for a number of representative days against the flow charts for those days using The Graphical Integration procedure (page 3-18).

PRESSURIZED FLOW OR FULL PIPE MEASUREMENT

Use

A magnetic flowmeter is used for pressurized flow or full pipe measurement and operates on a principle similar to that of an electric generator.

Basis of Operation

The basis of operation is that a conductor, in this case sewage or sludge flowing in a pipe, which crosses an imposed alternating magnetic field will generate a voltage proportional to the flow rate. Housed within the magnetic flowmeter is the alternating magnet and an electrode pair that receives the generated voltage. This voltage, appropriately scaled for the size of pipe, is displayed on the recorder as flow rate. No separate linearizing function is required for a magnetic flowmeter.

Calibration

Generally, a magnetic flowmeter cannot be checked out by an operator unless an independent means of gauging the flow rate exists (i.e. an open channel measuring device elsewhere in the plant). They are, however, a relatively realiable device and calibration once per year by a competent serviceman should be sufficient to ensure proper operation.

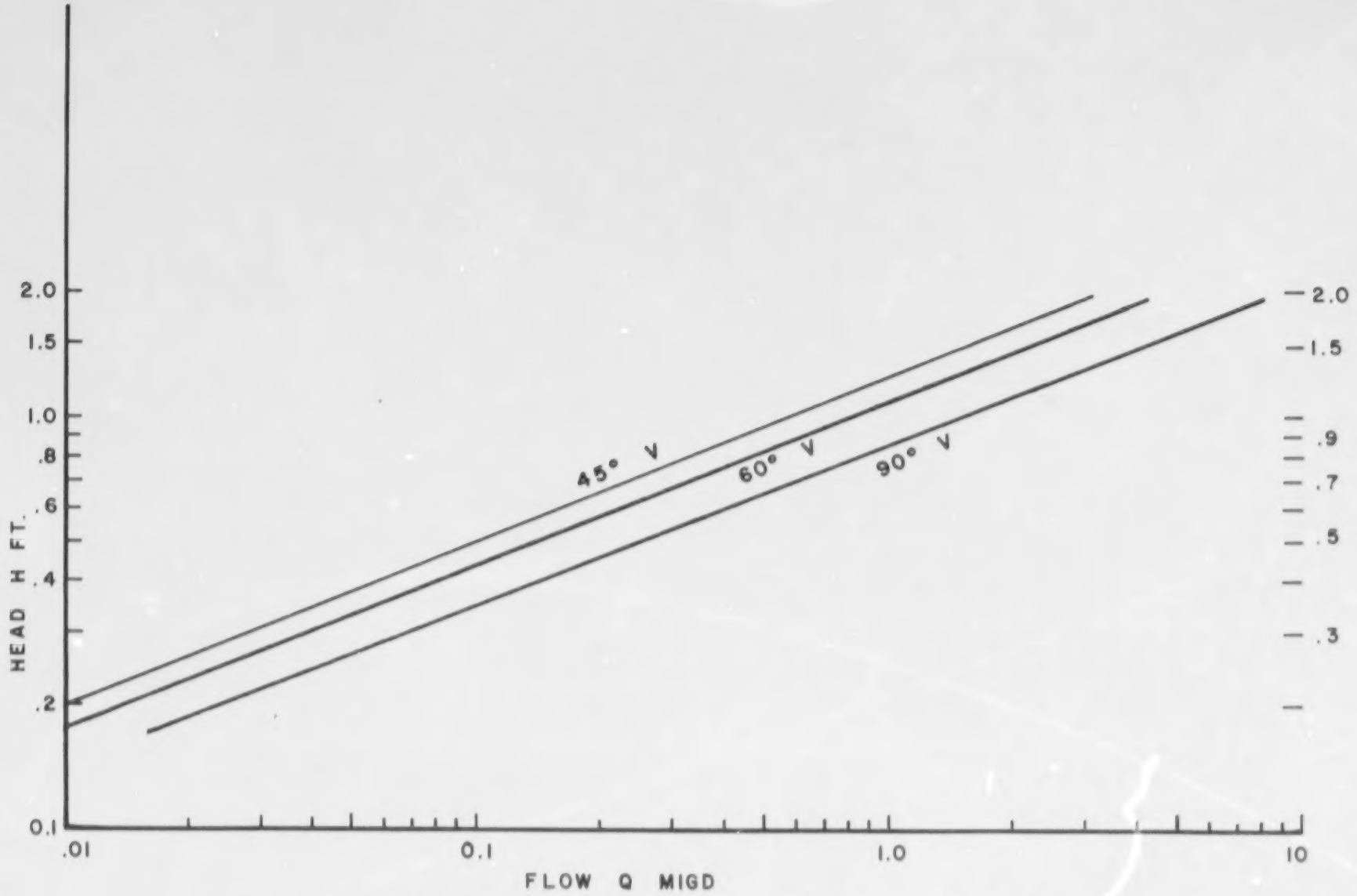


FIG.3-5 V-NOTCH WEIR CALIBRATION CURVES.

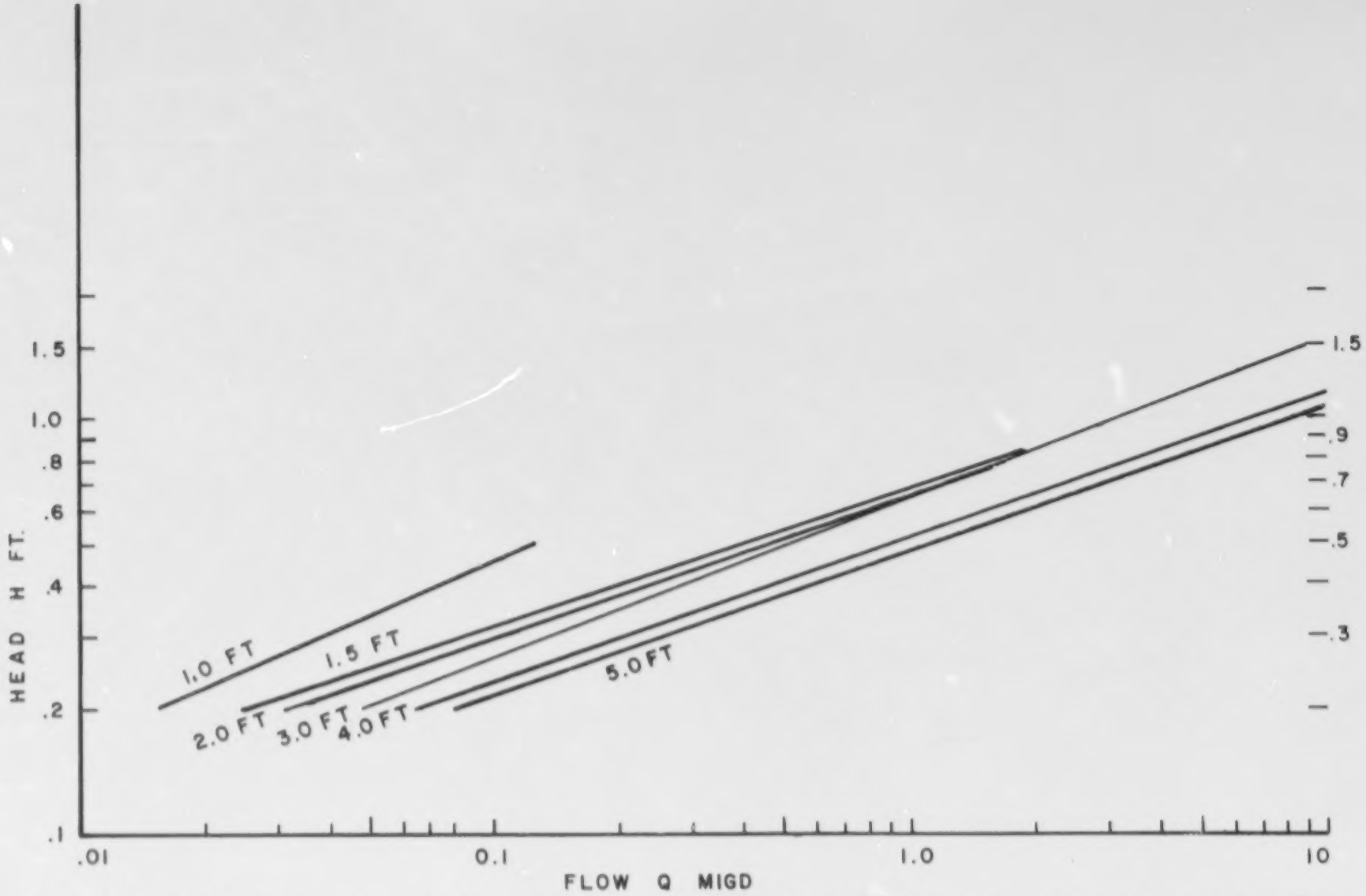


FIG. 3-6 RECTANGULAR WEIR WITH END CONTRACTIONS. CALIBRATION CURVES.

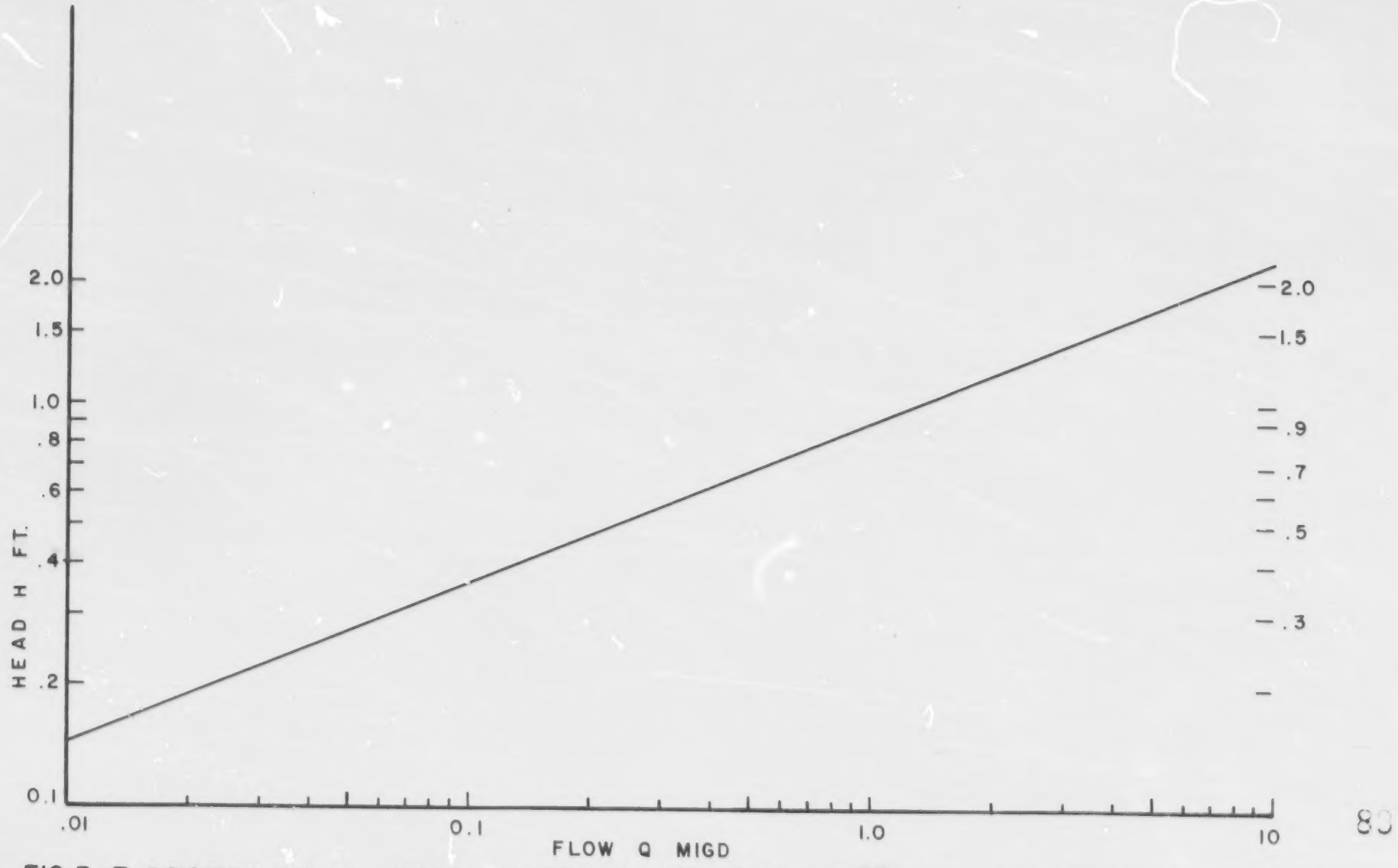


FIG. 3-7 RECTANGULAR WEIR WITHOUT END CONTRACTIONS. CALIBRATION CURVES.

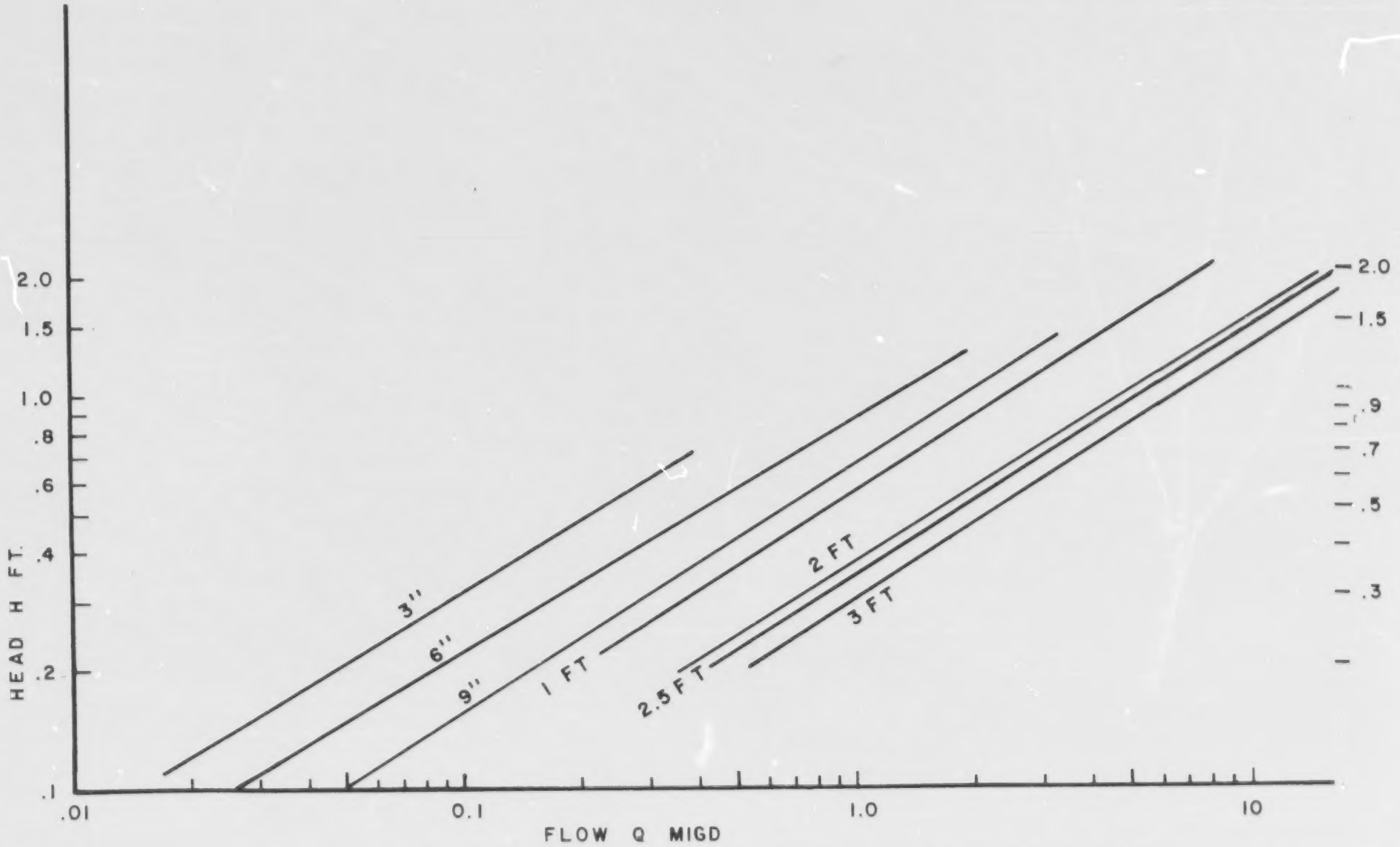


FIG. 3-8 PARSHALL FLUME. CALIBRATION CURVES.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 4

INFORMATION REQUIREMENTS

OBJECTIVES:

The trainee will be able to:

1. List six tests commonly used for process control.
2. List nine items of information required to carry out successful plant operation.
3. Recall the optimum sludge depths in the primary and secondary clarifiers.
4. Recall the application to process control of the results/calculations from:
   a. DO test.
   b. Solids settling test.
   c. MLSS determination.
   d. Sludge wasting calculations
   e. Oxygen Uptake Rate determination.
   f. pH measurement.
   g. F/M ratio.
   h. MOE laboratory tests.

INFORMATION REQUIREMENTS FOR PROCESS CONTROL

GENERAL

The processes within a plant, particularly biological processes, require constant and careful attention if they are to perform efficiently, and in some cases, if they are to perform at all. All the processes used in a sewage treatment plant (sedimentation, conversion of dissolved matter to particulate matter, conversion of particulate matter to gases and inert products) occur in nature. The difference is time: the plant does in hours what occurs in nature in the course of many days. Because these functions are made to perform more rapidly, some method of controlling processes to obtain maximum efficiency from the plant is necessary.

The only analytical data available to an operator on any specific day are those obtained from in-plant tests. Those normally done in the plant depend on the equipment available and the experience of the operating personnel. Regardless of the sophistication of equipment and personnel available, only those tests necessary and relevant to controlling and assessing the processes should be carried out. Tests most commonly performed are:

1. DO
2. Settling Test
3. Suspended Solids
4. Total Solids
5. pH Measurement
6. Oxygen Uptake Test

Other tests can be done by the Ministry laboratories on behalf of a plant to evaluate the performance of the plant and to estimate the effect of effluent discharge on the receiving stream. The most common analyses on plant influent and plant effluent are suspended solids, biochemical oxygen demand (BOD), total Kjeldahl nitrogen and total phosphorus.

Because of the uncontrollable time delay in receiving results from the laboratory, it is not possible to use the results obtained to operate the process. On a long-term basis, these results are used to monitor the process performance, verify in-plant tests and, generally, to accumulate accurate analytical data used to assess increasing BOD loads. These results can be recorded and used when dealing with future plant expansions. Procedures for requesting MOE laboratory assistance are at Appendix A.

The information obtained from in-plant test results and calculations by the operator or laboratory technician is used to control the waste treatment process on a day-to-day basis. In-plant test results should be used to regulate raw sludge to be pumped, activated sludge wasting, return sludge rates, liquid submergence in mechanically aerated plants, blower or compressor speeds, number of blowers in use in diffused air plants, chlorination rates, even by-passing of the aeration tanks to preserve the activated sludge system until a particular problem clears.

INFORMATION REQUIREMENTS

Data from tests is analyzed and interpreted for day-to-day operational control of the activated sludge process. The information required includes:

1. Sludge Depth by use of a probe
2. Sludge Settling Characteristics
3. Mixed Liquor Suspended Solids Concentration (MLSS)
4. Sludge Volume Index (SVI) using the settling test results and MLSS
5. Sludge Wasting Rate
6. DO
7. Oxygen Uptake Rate (Respiration Rate)
8. pH
9. F/M Ratio

Other data is also important to plant operations but it is generally specific to an individual plant.

Sludge Depth Probe

Various home-made devices are used for the measurement of sludge depths in primary and secondary clarifiers. There are also a number of electronic devices, utilizing a light source and photo-electric cell, available from equipment suppliers. These devices are simply lowered into the clarifier until a sound or visual signal indicates that the sensor head has reached the sludge water interface. Knowledge of the sludge depth in either the primary or secondary clarifier is handy since (if frequent checks are carried out) the operator can detect increasing sludge depths, and adjust sludge pumping rates before solids are lost from the clarifiers. Sludge settling tests are conducted on the mixed liquor suspended solids (aeration tank contents) as a further guide to clarifier performances and activated sludge conditions.

The operation of primary clarifiers should maintain 6-7 feet of relatively clear liquid above the sludge blanket. Under these conditions, a periodic hydraulic surge will not carry excessive solids into the aeration tanks.

Similarly, in the final clarifiers the sludge blanket should be kept below 4 feet in depth.

Sludge Settling

One of the tests most commonly done on mixed liquor (aeration tank contents) is the 30 minute settling test in a 1 litre (1000 ml) graduated cylinder. See Topic 9. The results obtained are presented either as a volume expressed in millilitres (ml), or as a percentage of the 1 litre volume.

During the first five or ten minutes of the settling period the activated sludge should be watched for settling characteristics. A slow settling sludge will produce a clear effluent but may not allow enough time for separation in the

clarifier. This can result in floc being carried over the weirs. Very fast settling sludge, on the other hand, tends to leave small particles in suspension, producing a cloudy effluent.

An increase in settled sludge volume (above normal) indicates, since sludge concentrations are increasing, more sludge wasting is necessary, or the sludge quality is deteriorating. More testing of solids concentrations and microscopic examinations of the activated sludge are therefore necessary.

In order to maintain a steady MLSS in the aeration tank, the settled organisms must be recirculated from the final clarifiers.

Mixed Liquor Suspended Solids Determinations (MLSS)

Mixed Liquor Suspended Solids determinations are used to control the quantity of activated sludge available to stabilize the organic matter in the incoming sewage.

Analysis for suspended and volatile suspended solids is described in Topic 10. In controlling plant processes suspended solids are sufficient because it can be assumed that the volatile portion will remain a constant proportion of the total suspended solids. However, *volatile suspended solids* should be used for comparison between plants *especially where one plant has phosphorus removal facilities.*

Results can be used in combination with other data for such things as calculating the sludge volume index (SVI) and the food to microorganism ratio (F/M ratio). A typical calculation is shown in Topic 10.

At smaller plants a *centrifuge* is used to estimate mixed liquor suspended solids concentrations. The centrifuge reading (usually of a 15 ml sample) of the volume occupied by the solids is compared to a *calibration curve*. The calibration curve is prepared by plotting centrifuge readings against suspended solids determined by weighing. Since this

method assumes a constant density of the compacted sludge which is rarely achieved, even a recently prepared calibration curve may be in error by 30 per cent.

Sludge Volume Index (SVI)

The Sludge Volume Index (SVI) is used to indicate the condition of sludge (aeration solids or suspended solids) for settling in a secondary or final clarifier. The SVI is the *volume* in ml occupied by one gram of mixed liquor suspended solids after 30 minutes of settling. It is a useful test to indicate changes in sludge characteristics.

Settled sludge volume while it is dependent on sludge quality is also a function of the mixed liquor suspended solids concentration. In order to eliminate the dependency of the settled volume on the solids concentration, the sludge volume index is used. It is calculated using the formula:

$$SVI = \frac{\text{30 min. settling test result (ml) x 1000}}{\text{MLSS (mg/l)}}$$

The proper SVI range for a plant is determined at the time when the final effluent is in the *best* condition regarding solids and BOD removals and clarity.

In general, good settling sludge has an SVI of 75-100; however many plants can operate quite well at SVI values as high as 250.

If SVI values are high, filamentous organisms may be developing in the sludge. The microscope can be used to determine what actions should be taken, such as adjusting conditions to prevent further filamentous growth, or chlorination to remove the organisms completely.

Sludge Density Index

The Sludge Density Index (SDI) is used in a way similar to the SVI to indicate the settleability of a sludge in a secondary clarifier or effluent. The calculation of the SDI requires the same information as the SVI test.

$$SDI = 100/SVI$$

Sludge Wasting

In order to maintain the desired F/M ratio in the plant, it will be necessary to waste some activated sludge. Ideally, this is achieved by a continuous, proportional wasting from the sludge return system. If continuous wasting is not feasible, batch wasting at periods of low hydraulic loads will attain the same results. The amount of sludge to be wasted is generally found by trial and error, but it can be calculated if the suspended solids in the aeration tank and sludge return are measured, and the following formula applied:

$$X = \frac{(Mo - Ml)\ V}{R}$$

Mo = MLSS (mg/l)
Ml = Required MLSS (mg/l)
V = Aeration tank capacity in gals.
R = SS in sludge return (mg/l)
X = Gallons to waste

FOR EXAMPLE:

1. Given the MLSS = 4000 mg/l, the SS in the sludge return is 9000 mg/l, and the aeration tank capacity is 300,000 gals. Calculate how much sludge return must be diverted to waste to reduce the MLSS to 3000 mg/l.

$$X = \frac{(4000 - 3000)\ 300{,}000}{9000} \text{ gal}$$

$$= \frac{1000 \times 300{,}000}{9000} \text{ gal}$$

$$= \frac{300{,}000}{9} \text{ gal}$$

$$= 33{,}333 \text{ gal.}$$

2. Calculate the continuous rate of wasting for a 24-hour period if volume to be wasted is 36,000 gallons.

$$\text{The Wasting Rate} = \frac{\text{gallons to be wasted}}{24 \text{ hours}}$$

$$= \frac{36{,}000}{24} \text{ gal/hr}$$

$$= 1{,}500 \text{ gal/hr}$$

DO Measurements

Dissolved Oxygen determinations are performed regularly on aeration tank contents to ensure that sufficient oxygen is being supplied to permit *aerobic* stabilization of organic matter.

There are various methods for determining dissolved oxygen. The Winkler Method (including the Hach DO Kit) and use of the DO Meter are discussed in Topic 8.

In an activated sludge plant, reasonable levels (1-2 mg/l) of dissolved oxygen must be maintained in the aeration tank(s) and final clarifier (s). DO determinations are required and samples must be taken in these tanks. The sample in the aeration tank is taken at the effluent end of the aeration system. The final clarifier sample is taken from the surface of the clarifier close to the effluent weir, not from the effluent launder.

Dissolved Oxygen may be determined at other sampling points such as the raw sewage or primary effluent but, since little or nothing can be done to change the values, the results are mainly for the record to help indicate any trend in the incoming wastes. Because these DO tests have little significance in plant control, they can be done infrequently.

Results of DO determinations will provide the operator with information from which:

1. He may increase aeration to offset low DO levels caused by increased flows, an influx of industrial waste or a change in composition of the influent.
2. He may decrease aeration to economize if DO levels are excessively high.

Oxygen Uptake Rate

Biological waste treatment in the activated sludge process is based on the ability of micro-organisms to utilize dissolved oxygen in breaking down soluble organic substances.

The oxygen uptake test is a means of measuring the respiration rate of the organisms in the activated sludge process. Since it measures the oxygen used in the process, it is a useful tool in the evaluation of process performance, aeration equipment and biodegradability of the waste. So that comparisons can be made between various plants, it is usually expressed as the specific uptake rate (SUR); i.e. the amount of oxygen in mg/l utilized by one gram of the volatile suspended solids in the activated sludge, in one hour. Once the operator has established the SUR for his own process, he can then recognize some upsets which may occur even before the effluent quality is affected.

This technique will give an operator some control and knowledge of his biological process and possibly of accidental spillages to the sewer coming from a particular company or industry. The accumulated SUR data combined with other operating data are invaluable to staff in assessing plant performance and capability.

Although not of direct interest to the plant operator, the oxygen uptake test can be used further in waste treatment technology.

1. Measurement of the oxygen uptake rates in conjunction with power measurements on the aeration equipment allows the calculation of aeration device efficiencies. This is known as the "Steady State Method" of aerator evaluation and is used in design and process evaluations.

2. In conjunction with laboratory or pilot scale aeration tanks, respiration rates can be used to assess the biodegradability of industrial wastes or the effect of industrial wastes on an existing system.

The procedure for determining oxygen uptake rate is dealt with in Topic 13. In order for the test results to be of any use in plant operation, the "normal" operating value must be known. For example, if the oxygen uptake rate (or

respiration rate) is normally 20 mg O_2/l/hr, it would be "normal" for it to vary from 10 to 30 mg O_2/l/hr during the day. However, if the uptake rate were suddenly to drop to 5 mg O_2/l/hr and the aeration tank DO increase, it would mean that an inhibiting (toxic) material had entered the system. Similarly, if the respiration rate rose to 60 mg O_2/l/hr with a marked drop in DO, it would indicate that a high organic load was being applied to the process which could adversely affect the effluent quality if it continued for any length of time.

In the case of a marked increase in respiration rate, a temporary solution might be an increase in DO; this could be done either by operating more compressors in the diffused air systems, or by adjusting the mechanical aeration equipment to obtain maximum DO. At the same time, an investigation should be made to determine the cause and origin of the increase in organic loading. The low uptake rates and high DO levels indicate inhibitions, either by adverse pH or toxic metals entering the system. Again, an effort to determine the source should be made, and the situation reported to the engineer in charge. *Enforcement of local by-laws should prevent the discharge of unsuitably high or low pH wastes.*

Respiration rates or uptake rates vary depending on the aeration tank solids concentration (MLSS); to eliminate this variable the rate is often expressed as the Specific Uptake Rate. The Specific Uptake Rate (SUR) is the oxygen utilized in one hour by one gram of mixed liquor *volatile* suspended solids (See Topic 13).

pH

Procedures for determining pH are covered in Topic 12.

The activated sludge process requires a pH range of 6.5 to 8.5, which is the usual range for domestic sewage. Any values above or below this range are a good indication that an alkaline or acidic industrial waste has been discharged into the sewer. Typical pH values for some common industrial wastes are shown in Figure 4-1, page 4-13.

Any prolonged variation from the normal pH range will result in process upset or failure. The operator can control pH to some degree by the use of chemicals. The only suitable remedy is enforcement of the industrial waste by-law by the proper authorities.

1. *Extremely high or low pH values may indicate "slugs" of industrial wastes have been discharged to the sewage system.*
2. *If the pH is less than 6.5, strong acid wastes are entering the plant.*
3. *If the pH is greater than 8.5, strong alkaline wastes are entering the plant.*

F/M Ratio

As discussed in Topic 2, microorganisms respond best when the F/M ratio is between 0.25 and 0.33. To determine this ratio, the total weight of BOD in the daily sewage flow, which is the food (F), is compared to the total weight of suspended solids (M) in the aeration. Since volatile solids form an almost constant proportion of the aeration suspended solids for a given plant, mixed liquor suspended solids (MLSS) can be used as the total weight of suspended solids (M).

$$\frac{\textit{Food (F)}}{\textit{Microorganisms (M)}} = \frac{\textit{Total weight of BOD in daily sewage flow (lb/day)}}{\textit{Total weight of volatile suspended solids in the aeration section (lbs) (MLSS)}}$$

The determination of MLSS is discussed in Topic 10, Analysis for Suspended and Volatile Suspended Solids. BOD is determined by either in-plant testing, or from results of tests conducted by the MOE laboratories at the request of the plant operator.

Examining the above equation, it is obvious that:

1. If the F/M ratio is too low for the type of plant, the lbs MLSS in the aeration tank is too high for the incoming BOD. This could be corrected by increasing the aeration rate and/or by reducing the sludge return rate.

2. If the F/M ratio is too high, there are insufficient microorganisms to adsorb the incoming organic material. This could be corrected by increasing the sludge return rate, increasing retention time.

EXAMPLE:

Given a flow of 3 MIGD, a primary effluent BOD of 150 mg/l, mixed liquor volatile suspended solids (MLVSS) of 1500 mg/l and an aeration tank volume of 1 million gallons, calculate the F/M ratio.

1. Calculation of BOD

From the BOD data received over a period of 6-8 months (determined by either in-plant testing or that done by MOE laboratories on behalf of the plant) and with corresponding flow values, the pounds of BOD applied to the plant per day can be calculated.

a) In the example the primary effluent BOD is 150 mg/l.

Since parts per million = ml per l

or = gms per million gms

or = lbs per million lbs

$\therefore$ 150 mg/l = 150 ppm

= 150 lbs/1,000,000 lbs

b) Since the flow is 3 MIGD and 1 gal weighs 10 lbs

$\therefore$ the flow in pounds/day is 30,000,000 lbs.

c) In each million pounds of flow we have 150 lbs of BOD

$\therefore$ In 30,000,000 lbs per day we have -

$$\frac{150 \times 30,000,000}{1,000,000}$$

= 4500 lbs/day BOD

2. Calculation of MLVSS

Calculation of MLVSS is dealt with in Topic 4. In this problem we are given that MLVSS is 1500 mg/l.

a) Remember 1(a) above

1500 mg/l = 1500 lbs/1,000,000 lbs.

b) Since the volume of the aeration tank is given as 1,000,000 gallons, we must convert the volume to lbs. (i.e. multiply by 10 since 1 gal weighs 10 lbs). The weight of material in the aeration tank therefore is 10,000,000 lbs.

c) The total weight of MLVSS in the aeration tank therefore is $\frac{1500 \text{ lbs}}{1{,}000{,}000 \text{ lbs}} \times 10{,}000{,}000 \text{ lbs}$

$= 15{,}000 \text{ lbs}$

3. Calculation of F/M Ratio

$$F/M = \frac{\text{lbs BOD/day}}{\text{lbs MLVSS}}$$

$$= \frac{4500}{15{,}000}$$

$$= 0.3$$

4% CAUSTIC SODA	14
INDUSTRIAL CLEANING WASTES	13
TANNERY WASTES	12
	11
MILK OF MAGNESIA	10
TEXTILE & LAUNDRY WASTES	9
DOMESTIC SEWAGE	8
PURE WATER	7
CHEESE WASTES	6
DISTILLERY WASTES	5
	4
ORANGE JUICE	3
STEEL PICKLING WASTES	2
	1
5% SULPHURIC ACID	0

ALKALINE

ACIDIC

Figure 4-1 pH VALUES OF INDUSTRIAL WASTES

MINISTRY LABORATORIES

TESTING

The forms at pages 4-15 and 4-16 include instructions on the information to be included when requesting an analysis by the Ministry Laboratories. Preliminary discussions with the laboratory staff may be necessary to ensure an appropriate and reliable analysis.

Routine chemical analyses are completed at the Main and Regional Laboratories within a maximum of two weeks, although the majority of results are returned to the plant in a much shorter period. Initial delays in forwarding the sample could add to this time interval, and may also cause the samples to deteriorate before analyses. *All samples should be sent out promptly.*

The results included on the Ministry of the Environment lab test sheets will also provide a periodic check on the MLSS analyses run at the plant, as well as the volatile contents of the MLSS. If results indicate that volatile content at the constant F/M ratio is dropping markedly over a period of time, inorganic material may be entering the plant. In addition to knowing the final effluent quality and being able to compare this with the Ministry of the Environment objectives, the nutrient input and removal in the plant can also be determined as required by a plant phosphorus removal program.

Occasionally, the Ministry will request some additional analyses of the raw sewage samples for such things as heavy metals or cyanides of the raw sewage samples since high levels (above 2 mg/l) of these materials will reduce the activated sludge treatment efficiency.

MAIL ADDRESS
MINISTRY OF THE ENV[illegible]NMENT
LABORATORY BRANCH
P.O. BOX No. 213
REXDALE, ONTARIO

MINISTRY OF THE ENVIRONMENT
LABORATORIES

EXPRESS ADDRESS
RESOURCES ROAD
HIGHWAY 401 & ISLINGTON AVE.
TORONTO, ONTARIO

Municipality .....
Source ..... Region .....
Program: .....
Date sampled ..... by .....
Date analysed ..... Date reported .....

Report to: .....
Address: .....

C	
Bi	
Ba	

LAB NUMBER	SENDER'S NUMBER	SAMPLING POINT LOCATIONS AND TIME	NATURE OF SAMPLE, DANGEROUS CONSTITUENTS, PRESERVATIVES USED, COMPOSITING DATA, ETC.	✓CHECK BELOW IF CHLORINE PRESENT

LAB. NUMBER											

4-15

101

102

12-084

SAMPLING DIRECTIONS

INSTRUCTIONS

This form should accompany all samples, with copies included for each laboratory (chemical, bacteriological and biological) to which samples are submitted. Samples for each laboratory should be taken in separate, individually labelled bottles. Suitable bottles are available, on request, from the laboratory shipping department. Phone 248-3027. No charge is made for routine analyses. The sender shall bear all shipping costs.

ALL SAMPLES ARE PERISHABLE. Please submit promptly, as early in the week as possible. Keep samples cool, preferably chilled, and away from light. Avoid freezing. Samples may be delivered to the laboratory anytime, at night, or on weekends and holidays, they will be refrigerated.

BACTERIAL

STERILE BOTTLES (6 OZ. OR MORE) MUST BE USED. TO ENSURE RELIABLE RESULTS, SAMPLES SHOULD ARRIVE AT THE LABORATORY WITHIN 24 HOURS OF SAMPLING OR BE REFRIGERATED IF DELAY IS UNAVOIDABLE.

For other than routine examinations, please enquire beforehand. Phone 248-3003. DO NOT OPEN THE BOTTLE until all other sampling preparations have been made, then discard the cellophane dam. Samples from taps must be taken only after aerators, screens and hoses, etc., have been removed, and THEN only after the water has been running for two minutes. Samples from open waters must be taken by tying a clean copper wire to the bottle's neck before removing the cap, and then lowering it below the water's surface. Collect samples directly in the sterile bottles provided and NOT BY MEANS OF A DIPPER. While bottle is being filled, hold cap in hand — DO NOT LAY IT DOWN. Do not touch its inner surfaces or the mouth of the bottle with hands or any other object. Always leave an air space in the bottle unless advised otherwise.

CHEMICAL

Our standard 32-oz. bottle contains sufficient sample for routine tests only. (Since sludge samples develop gas, leave bottle half-empty.) Additional non-routine tests require more sample (2 bottles) and special sampling and handling techniques. Please enquire beforehand. Phone 248-3027.

BIOLOGICAL

Any specimens for identification should be refrigerated or preserved in 5% formaldehyde and submitted as soon as possible. Where special biological analyses are desired, sampling procedures are best arranged with the biological staff beforehand. Phone 248-3011.

SAMPLE CLASSIFICATION

A variety of samples is received. To ensure that your samples receive the proper tests, CLASSIFY EACH SAMPLE AND CHOOSE THE ANALYSIS DESIRED, BELOW.

WATER

These include samples taken from sources of supply, treatment and distribution systems and waters used for drinking, domestic or industrial purposes.

☐ BACTERIAL Coliform ☐ CHEMICAL—Hardness, Alkalinity Iron, Chlorides, pH ☐ BIOLOGICAL EXAMINATION

SEWAGE AND STREAM

Include samples taken from waste discharges, waste collection and treatment systems. To detect pollution, the probable source should be sampled, together with the receiving water, prior to and following entry of the pollution. Describe the type of pollution, and its ingredients, below.

☐ BACTERIAL Coliform ☐ CHEMICAL—Biochemical Oxygen Demand, Suspended Solids or Turbidity ☐ BIOLOGICAL EXAMINATION

SPECIAL ANALYSES

For other than routine determinations, preliminary discussion with laboratory staff facilitates and often is essential for an appropriate and reliable analysis. Phone 248-3421. Enter below a brief description of the problem which has necessitated the submission of samples, designating the materials sought or suspected of being present.

Describe Problem: _____

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 5

IDENTIFICATION AND SOLUTION OF OPERATING PROBLEMS

OBJECTIVES:

The trainee will be able to

1. Name the information required to identify and solve major causes of plant upsets.

2. Interpret in-plant and laboratory test data and apply this information to the solution of operating problems.

3. List three major outside sources of plant upsets and the possible steps that can be taken to rectify the problems.

PLANT OPERATING PROBLEMS AND SOLUTIONS

INTRODUCTION

It is not possible to lay down a rigid procedure for evaluating the performance of a water pollution control plant or for problem solving at a plant. There are, however, five basic pre-requisites which are necessary in order to identify and solve operating problems:

1. A complete knowledge of the activated sludge process.
2. A good working knowledge of the design criteria and capacities of the treatment system.
3. An ability to recognize the problem exists.
4. The ability to identify the source of the problem.
5. The knowledge to solve the problem.

INVESTIGATION INTO OPERATING PROBLEMS

An investigation into operating problems, whether carried out by the operator or by an inspector visiting the plant, can be conducted in a similar manner. This procedure can be divided into three main areas as follows:

1. A discussion with other operating staff or superintendent.
   - A visual inspection of the plant during which such things as the colour of the mixed liquor, quality of the final effluent, odours from the system, and overall appearance of the plant can be taken into consideration. At this time an experienced operator should be capable of identifying any incorrect procedures or operating techniques which are being used and likely to cause problems.

2. A review of operating records.
   - Flow records should be examined. Flow variation should be noted and flow data should be reviewed further and compared with design flow, the type of system, and the population of the town. A manual check of flow measuring equipment (the head across the primary measuring element compared with the chart) should always be made. Many plants inspected by staff of this Ministry have had as high as 45% error between actual flow and recorded flow.
   - Records of in-plant tests should be examined to observe the consistency in operation and any trends that are apparent which may have lead to the current problem.

3. Sampling procedures should be checked.
   - It is important to note the type of samplers or sampling procedures being used, the times at which the sampling is carried out, the location, and the frequency. It is worthy of note that even with the world's best chemist, incorrect sampling procedures will result in useless and erroneous data.

4. Analytical Results.
   - Result sheets should be inspected and reviewed for unusual strength wastes, wide pH fluctuations and other abnormal data eg: soluble toxic metals.

CAUSES OF PLANT UPSETS

There are three major outside sources of plant upsets. These are:

1. Hydraulic Loading
2. Organic Loading
3. Toxic Wastes

HYDRAULIC LOADING

Flows may be experienced which are either in excess of or extremely below design capacity. These flows may be caused by:

1. Infiltration during periods of above normal rainfall;
2. Improper operation of a main sewage lift station caused by frequent starting and stopping of pumps;
3. Illegal sewer connections;
4. Exfiltration because of broken or leaking sewers.

Excessive Hydraulic Loading may result in:

1. Flooding of weirs and superstructures;
2. Poor effluent because of carryover of solids due to excessive clarifier loadings;
3. Washout of the mixed liquor from the aeration tanks and a reduction or breakdown in the process.

Inadequate Hydraulic Loading may result in:

Inadequate food for microorganisms (low F/M ratio) and a depletion of oxygen in the settling tanks.

Indicators

Indications that problems may be imminent or exist because of hydraulic loading are obtained from:

1. Flow meter readings or by observing the level of flow on the walls of the channel;
2. Odours and colour of influent;
3. Scum blanket in wet well;
4. Excessive grit;

5. Changes in MLSS;

6. Change in DO;

7. Clarifier upflow rate excessive.

Corrective Actions

1. In the case of excessive hydraulic loads overloading the secondary system, bypass a portion of the flow from the primary system to a holding tank if available and pump holding back into system during low flow condition;

2. Increase aeration to increase MLVSS if excessive flow;

3. Increase sludge wastage rate if reduced hydraulic load, thus obtaining an acceptable F/M ratio.

ORGANIC LOADING

Problems Arising

If there is high organic loading in the secondary system, particularly of material requiring a rapid oxygen uptake, a reduction or depletion of oxygen will occur and the treatment efficiency will drop. Soluble solids and solids with low settling rates will be carried over into the clarifier(s) because of poor treatment efficiency. Sludge bulking will occur in the final clarifier(s). Floating matter will be discharged in the effluent.

Indicators

Problems may be imminent or exist because of organic loading if:

1. DO values significantly changed;

2. Odours of hydrogen sulphide exist;

3. Low results are obtained from settling test;

4. SVI is high;
5. There is rising sludge in clarifier;
6. F/M ratio is excessive;
7. Uptake rates are high;
8. There is extreme sludge growth (if the dissolved components have a high BOD).

Corrective Actions

Possible corrective actions may include:

1. Increase sludge return rate to adjust F/M ratio;
2. Chlorinate the return sludge if microscopic examination reveals filamentous growth;
3. Increase aeration to adjust F/M ratio;
4. If possible, reduce loading to aeration tank(s);
5. If possible, increase retention time in aeration tank(s).

TOXIC WASTES

Problems

The possible effects of excessive discharges of toxic wastes are:

1. A reduction in biological treatment efficiency or a complete breakdown in the process;
2. Corrosive problems throughout the plant;
3. Odours such as hydrogen sulphide or a characteristic antiseptic smell;
4. Poor settling.

Indicators

Indications that problems may be imminent or exist because of industrial wastes are obtained from:

1. Odour and colour of influent;
2. Reduction or complete breakdown of the biological process;
3. Unusual amounts of solids in influent;
4. Reduced settling rates determined by test;
5. Low DO;
6. Unacceptable pH and F/M ratio;
7. Filamentous growth in mixed liquor determined by microscopic examination;
8. Rising sludge in final clarifier(s);
9. Temperature change.

Corrective Actions

Possible corrective actions are limited. The problem can normally only be cured by locating the source of the toxic waste and eliminating it, either by by-law enforcement or installation of a treatment process at the plant source. In certain instances it may be possible for the plant operator to:

1. Bypass the biological treatment process as soon as the problem has been detected, and
2. If the process has been contaminated, dispose of the floc and contaminated sludge and restart the system. This is a most drastic step and not usually required.

SUMMARY

Although there are not too many process changes that an operator can make to overcome plant upsets, a thorough knowledge and understanding of the plant design characteristics and his normal operating process characteristics is essential if prompt and effective action(s) are to be taken. Generally the corrective actions, based on observations and analysis of test results, involve changes in one or more of the following:

1. Degree of aeration;
2. Return sludge rate;
3. Sludge wastage rate;
4. Concentration of MLSS;
5. Retention time if spare tank capacity is available;
6. pH control by use of chemicals;
7. Chlorination of return sludge.

The principal objective of the operator's actions is to return the process to optimum conditions by establishing a good F/M ratio and a proper DO content. In determining the actions that he takes, the operator must consider his visual observations, the results of in-plant tests and the design data relating to his plant.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 6

PHOSPHORUS REMOVAL

OBJECTIVES:

The trainee will be able to

1. Name three chemicals suitable for phosphorus removal.
2. Recall three possible application points for chemicals used in phosphorus removal.
3. List the effects that phosphorus removal chemicals have on the raw sludge concentration and the operation of anaerobic digesters.
4. Calculate the feed rate for chemicals used in phosphorus removal.
5. Recall five means by which the operator can control the phosphorus removal process.

GENERAL

In recent years the phosphates in wastewater treatment plant effluents have been identified as a major factor in the eutrophication (rapid aging) of receiving waters. Industrial waste discharges and run-off also contribute to this problem. Excessive amounts of nutrients (phosphorus, nitrogen, etc.) can cause the rapid growth of algae and weeds. Algae and weeds will settle to the bottom, decompose and use up the dissolved oxygen causing the destruction of the life cycle systems normally found in unpolluted lakes, rivers and streams.

Because it is a major cause of eutrofication and present technology provides a means to control it, phosphorus was the nutrient selected to be removed from plant effluents being discharged into Lake Erie, Lake Ontario, the Ottawa River system, and inland recreational areas. Phosphorus removal facilities have been installed in a number of wastewater treatment plants and future years will see an increasing number.

There are a number of ways to remove phosphorus. These include reverse osmosis, adsorption, ion exchange and chemical precipitation. Chemical precipitation using commercially available chemicals is the least costly, both from capital and operating costs, and is the system of choice in Ontario. It is the method discussed in this topic.

Source of Phosphorus

Phosphorus in the plant influent comes in many forms. It consists of organic phosphorus from food and wastes, polyphosphates from detergents, and precipitated orthophosphate from chemical reactions between metal ions in the wastewater and dissolved orthophosphates.

The concentration of the phosphorus in the wastewater is measured as -

1. Total phosphorus which includes all forms of phosphorus as mg/l P.

2. Soluble, reactive phosphates as mg/l P.

Because of the complexity of the test required to determine phosphorus in the influent or effluent, the tests are normally done by the Ministry Laboratories. The procedure is outlined in Topic 15 of this manual.

Mechanism of Removal

The mechanism of phosphorus removal is a combination of chemical and physical reactions which include the chemical precipitation of the soluble, reactive phosphates by the metal ions (Ca^{+2}, Fe^{+3}, Al^{+3}) introduced. Other important reactions are the formation of metal hydroxides which adsorb non-reactive phosphates and trap finely suspended material containing phosphates bound to organic matter. Sufficient time for flocculation and sedimentation of this combined floc is needed to produce an effluent with the desired low phosphorus concentration (<1.0 mg/l P). Good mixing at the point of chemical addition is also important. Rapid mixing followed by slow, gentle mixing before sedimentation will produce the best results. Sufficient clarifier (primary or secondary) detention time (over 2 hours) and low upflow rates (<800 gal/day/ft^2) at peak flows are also needed to achieve good clarification if chemicals are added for phosphorus removal.

CHEMICALS USED

Jar tests and possibly full scale pilot studies should be conducted before the best suitable chemical is selected. The following commercially available chemicals are normally used for phosphorus removal.

1.	Ferric Chloride	$FeCl_3$
2.	Ferrous Chloride	$FeCl_2$ (waste pickle liquor)
3.	Ferrous Sulphate	$FeSO_4$ (waste pickle liquor)
4.	Alum	$Al_2(SO_4)_3.14\ H_2O$
5.	Hydrated Lime	$Ca(OH)_2$

Of the chemicals listed above, alum, ferric chloride, and hydrated lime are most widely used although waste pickle liquor is gaining in popularity since a substantial cost saving can be realized. Waste pickle liquor should only be used in secondary treatment plants, because the 2-valent (ferrous) iron has to be oxidized to the 3-valent (ferric) iron in order to precipitate phosphates. To provide sufficient time and oxygen for oxidation, the point of addition of waste pickle liquor should be the influent end of the aeration tank. Handling, storage, bulk delivery, etc., are similar to ferric chloride.

Ferric Chloride

Ferric chloride is normally used in the liquid form although it is available in the dry form in drums. The reddish-brown liquid is corrosive and stains concrete. With proper dilution, fairly low temperatures can be tolerated. For outside storage in Ontario, heated, fibre-glass reinforced plastic storage tanks should be used. All other equipment used (pumps, feed lines, etc.) should be heat-treated and able to handle corrosive liquids since commercial ferric chloride solution (and pickle liquor) contains strong acid. The acid in solution and the acid produced when ferric chloride is added is normally neutralized by natural alkalinity in the wastewater. Additional alkalinity (lime, caustic soda) may have to be added to wastewaters with low alkalinity. The ferric chloride can be added to either raw sewage or in the secondary section. Experience indicates that the latter point of addition yields better results at lower costs. The ferric ions (Fe^{+3}) combine with the orthophosphate to produce a precipitate (iron phosphate) and with the hydroxyl ion to produce a floc (ferric hydroxide).

Alum

Alum is easier to handle than lime and is somewhat less corrosive than ferric chloride. It is usually purchased in liquid form, although it can be procured in 100 lb bags in dry form. The aluminum ions (Al^{+3}) combine with the

orthophosphate to form a precipitate (aluminum phosphate) and with the hydroxyl ions in the water to form a floc (aluminum hydroxide). It also produces an acid (sulphuric acid) which may be neutralized by the alkalinity available in the sewage or by added alkalinity.

Alum is delivered and stored in liquid form and as for ferric chloride, involves a large capital outlay for storage tanks and ancillary equipment. As alum crystallizes at fairly high temperatures, heating of tanks and feed lines is also necessary.

Alum can be added to either the raw sewage for phosphorus removal in the primary clarifiers, or in the aeration tank effluent. At most Ministry of the Environment secondary treatment plants, addition is made to the secondary section of the plant in the aeration effluent.

Hydrated Lime

Lime is employed because it is comparatively inexpensive. A portion of lime reacts with the orthophosphate to form an insoluble compound. The remaining lime and the magnesium either in the sewage or introduced in the lime form a floc causing the precipitated phosphates and other suspended solids to settle quickly. Lime also reacts with the CO_2 in the wastewater to form calcium carbonate.

Bulk lime is delivered in 10 or 20 ton loads and blown into a storage hopper or slurry make-up tank. The quantity normally required makes the use of fifty pound bags impractical. Dry storage works well, although problems can result unless the lime remains dry, there are no uncalcined pebbles, and if there is sufficient and constant water pressure for slurry make-up. Slurry storage involves a large capital outlay, unloading of the bulk lime is less than clean and if the slurry is not used quickly it will lose some of its effectiveness.

Lime should normally be added to the raw sewage ahead of the primary clarifier. Dosage can be most effectively controlled by maintaining the pH of the primary effluent at about 9.5. A lower pH will probably not produce the right conditions for the reactions to proceed quickly and effectively, the phosphorus being carried over with the solids in the effluent. A high pH (>8.4) could inhibit biological growth in the mixed liquor. The primary effluent can be low in BOD because of the additional removal of organic materials by the lime.

Lime is particularly suitable from an economic point of view in waters of low alkalinity. Despite handling difficulties, lime will produce an effluent from which most of the heavy metals have been removed by precipitation as hydroxides and which has been softened to some extent. In some areas, because of a combination of factors, lime is the only viable choice. Digestion of lime sludge appears not to be a problem.

PROCESS CONTROL PROBLEMS

Those likely to be encountered with ferric chloride, pickle liquor and alum include:

1. If added to the raw sewage:

   a) increased raw sludge removal is required because of increased sludge volumes and lower sludge solids concentration. Increased raw sludge volumes could cause digester problems due to hydraulic overload.

   b) The raw sludge may be acidic (pH < 7.0) and could cause problems with anaerobic digestion. Alkalinity (lime) may have to be added to the digester.

2. If added to the aeration tank:

   a) sludge return and sludge wasting have to be increased to prevent excessive sludge accumulations in the clarifier and to prevent the formation of a non-volatile, inert mixed liquor.

b) High dosages to the aeration tank could result in a mixed liquor with a low pH at which the precipitated phosphates may redissolve and biological growth may be retarded. Addition of alkalinity (lime, caustic soda) to the aeration tank will be necessary to counter this problem.

3. Feeding the chemicals at a constant rate (X ml/min) could lead to one or more of the aforementioned problems if extreme variations in daily flows are encountered at the plant. Pacing chemical addition according to incoming flows is therefore recommended.

4. Chemical addition for phosphorus removal usually results in increased removals of toxic heavy metals from the wastewater and this could result in high levels of heavy metals in the digested sludge and could make this sludge unsuitable for disposal on farm land.

Process control problems likely to be encountered with lime include:

1. The sludge produced, if high magnesium lime is used, tends to be fluffy and may float above the scraper mechanism of the clarifiers if it is allowed to build up. Normally, "High Calcium" lime does not give this problem.

2. The deposition of precipitate at points of turbulence and on all surfaces generally. Clarifier weirs and channels must be cleaned often and pipes flushed to prevent clogging. Recirculation of primary sludge will reduce this problem substantially.

3. Because the amount of sludge produced is greater, sludge must be removed from the primary clarifier more often.

4. pH control in the aeration tank. A close check must be maintained to keep the pH below 8.4 to prevent destruction of biological sludge.

5. Overdosing with lime may cause digester upsets.

SUMMARY

Table 6-1 summarizes the use of lime, alum and ferric chloride in phosphorus removal.

Table 6-1 CHEMICAL ADDITION FOR PHOSPHORUS REMOVAL

CHEMICAL	POINT OF ADDITION	COMMENT
Lime	Raw Sewage	Increased raw sludge concentrations and volumes, higher raw sludge pH. Primary effluent will have lower BOD, higher pH values. Maintain close check on aeration tank - pH should not go over 8.4.
Lime	Final Effluent (Tertiary System)	Additional clarifier needed. Problems with chemical sludge volumes.
Ferric Chloride Pickle Liquor Alum	Raw Sewage	Slight decrease in raw sludge concentrations possible, increased sludge volumes. Primary effluent BOD values lower.
Ferric Chloride Pickle Liquor Alum	Aeration Tank	Decrease in aeration tank volatile solids, increased activated sludge return and wasting required, resulting in changes in raw sludge concentration. Pickle liquor added to aeration influent.

DOSAGE

Control

Influent conditions cannot be used as a basis for determining the dosage required to produce the required effluent (1.0 ppm P) or 80% removal because:

1. Sewage is complex and variable mixture of organic and inorganic compounds.
2. Removal is not only a function of the completeness of chemical reactions but also of the degree of flocculation adsorption and sedimentation.

Dosage must be determined for each plant on the basis of experience gained from jar testing, full scale testing and recent operations. The procedure for phosphorus determination is described in Topic 15.

If the plant is not producing an effluent which meets the standard, the operator can control the process of phosphorus removal by employing one or more of the following:

1. Changes in dosage.
2. Sludge wasting.
3. Changes in pH by addition of lime or soda ash.
4. Investigate use of other chemicals.
5. Change point of chemical addition.

Calculations

In calculating chemical dosage, the operator must bear in mind that the active ingredient of the chemical added is only the metal ion; e.g. calcium (Ca^{+2}), aluminum (Al^{+3}), ferric (Fe^{+3}). One therefore calculates the amount of Fe^{+3} required to reduce the phosphorus and must then determine the amount of ferric chloride solution required which contains Fe^{+3}, acid and water. See Mathematics for Operators page 50.

Chemical Dosage Calculation for Phosphorus Removal

Examples:

In a plant with an average flow of 4.0 MGD ferric chloride is used at a dosage of 10 mg/l Fe^{+3} added after the aeration tanks. What ferric chloride flow rate measured in ml/min is needed?

Data: weight of ferric chloride is 14.1 lb/gallon

Ferric chloride contains 14.0% wt/wt Fe^{+3}

Ferric chloride contains 41.0% wt/wt $FeCl_3$

a) Step by Step Calculations:

$$10 \text{ mg/l } Fe^{+3} = \frac{10 \text{ lb } Fe^{+3}}{100{,}000 \text{ G}} \times 4{,}000{,}000 \text{ G/D} = 400 \text{ lb/D of } Fe^{+3}$$

therefore liquid ferric chloride ($FeCl_3$) needed =

$$\frac{400 \text{ lb/D } Fe^{+3}}{0.14} = 2857 \text{ lb/D}$$

hence volume of liquid $FeCl_3$ needed =

$$2857 \text{ lb/D} \times \frac{1 \text{ G}}{14.1 \text{ lb}} = 202.6 \text{ G/D}$$

hence ml/min of liquid $FeCl_3$ =

$$202.6 \text{ G/D} \times \frac{1 \text{ day}}{24 \text{ hr}} \times \frac{1 \text{ hr}}{60 \text{ min}} \times 4546 \frac{\text{ml}}{\text{G}} = \underline{\underline{639.7 \text{ ml/min}}}$$

b) Calculation by Formula:

$$\text{ml/min} = \frac{(\text{dosage mg/l})\ (\text{plant flow MGD})}{\left(\begin{matrix}\% \text{ active chemical} \\ \text{as fraction}\end{matrix}\right)\left(\begin{matrix}\text{weight of chemical} \\ \text{lb/gal}\end{matrix}\right)} \times (31.57) =$$

$$\text{ml/min} = \frac{(10 \text{ mg/l})(4. \text{ MGD})(31.57)}{(0.14)(14.1)} = \underline{\underline{639.7 \text{ ml/min}}}$$

SUBJECT: 2

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 7

MICROSCOPIC EXAMINATION OF ACTIVATED SLUDGES AND INTERPRETATION OF RESULTS

OBJECTIVES:

The trainee will be able to

1. Demonstrate how to use a microscope.

2. Use the microscope to:

   a) differentiate between the various species, and

   b) identify the filamentous organisms.

3. Classify conditions which have occurred, are occurring, or will occur within the activated sludge treatment process according to the defined protozoa.

4. Name the major components of a microscope, and demonstrate the maintenance required for proper functioning of the microscope.

MICROSCOPIC EXAMINATION OF ACTIVATED SLUDGE

FUNCTIONS OF MICROORGANISMS

General

Activated sludge contains millions of microscopic forms of life. The specific contributions made by each of these types of living organisms, however, in the purification of wastewater are not firmly established. Of interest from a process control point of view are bacteria, protozoa and rotifers. See Figure 7-1. There are other organisms, such as nematodes (roundworms) and waterborne insect larvae but these do not have any significance in the treatment process. See Figure 7-2. All of these living organisms require carbon in some form as energy, a small amount of which must be carbon dioxide; most also require some organic carbon compounds (such as sugars) and nitrogenous compounds as well as sulphur and phosphorus.

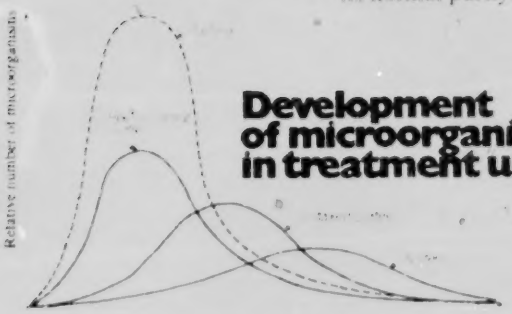
In the activated sludge process there are two forms of bacteria present, without which there would be no biological treatment as we know it. They are:

1. Autotrophs which require only carbon dioxide as a carbon source, and
2. Heterotrophs which require, in addition to carbon dioxide, the carbohydrates and other organic compounds produced by the action of the autotrophs.

These bacteria use the organic matter for food and reproduction. In turn the new bacterial growth is consumed by the protozoa (unicellular animals) and rotifers (a higher form of animal life which metabolizes solid food). The increased bacterial growth results in the accelerated extraction of wastes from solution, improved flocculation characteristics in the activated sludge and a biological floc with improved settling characteristics.

How microorganisms function in biological waste systems

Wastes contain many varieties of living organisms. Discussed here are some of the beneficial types which break down and transform pollutants into harmless substances, thereby helping to return water to its normal purity.



Development of microorganisms in treatment units.

Biological floc

I = Active solids
I + II = Volatile solids
III = Inert solids
I + II + III = Total solids

Bacteria

Bacteria are microscopic one-celled plants. They are responsible for the purification of the organic pollution contained in waste and form the active part of floc. The bacterial species present, such as pseudomonas, flavobacterium, etc., are dependent on the nature of the organic matter to be decomposed. They develop rapidly in all biological treatment systems.

Bacteria cell

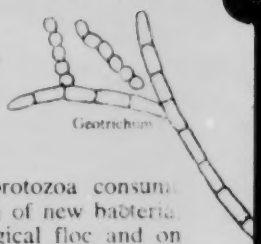
In a bacterial cell, the organic matter must cross the cytoplasmic membrane before being utilized.

Filamentous bacteria

These bacteria develop where carbohydrates are present and where there is low dissolved oxygen content. The result is bulking and poor settling.

Fungi

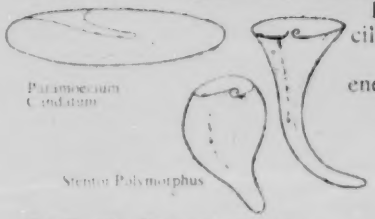
Like bacteria, these multicellular microscopic plants decompose soluble organic matter. Development is most rapid when the pH is acid and the oxygen level is low. Their presence is undesirable because they cause bulking.



Protozoa

Unicellular microscopic animals, protozoa consume bacteria, thus promoting the growth of new bacteria. They feed on the surface of biological floc and on dispersed bacteria, which results in a clear effluent. The presence of protozoa indicates that there is sufficient dissolved oxygen and a lack of toxic elements. There are two basic forms:

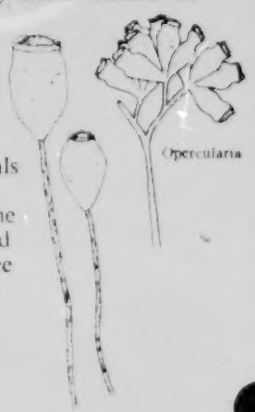
Free-swimming ciliate protozoa



Fine hairs allow these ciliates to swim rapidly. They have a high energy level and require a large quantity of organic food.

Stalked ciliate protozoa

Normally found in high-rate systems in equal numbers with the free-swimming ciliates, they attach themselves by their stalks to solid particles.



Rotifers

Multicellular microscopic animals which feed on bacteria and protozoa, rotifers exist only in the presence of dissolved oxygen and are an indication of a high degree of treatment. They are normally found in extended aeration systems.

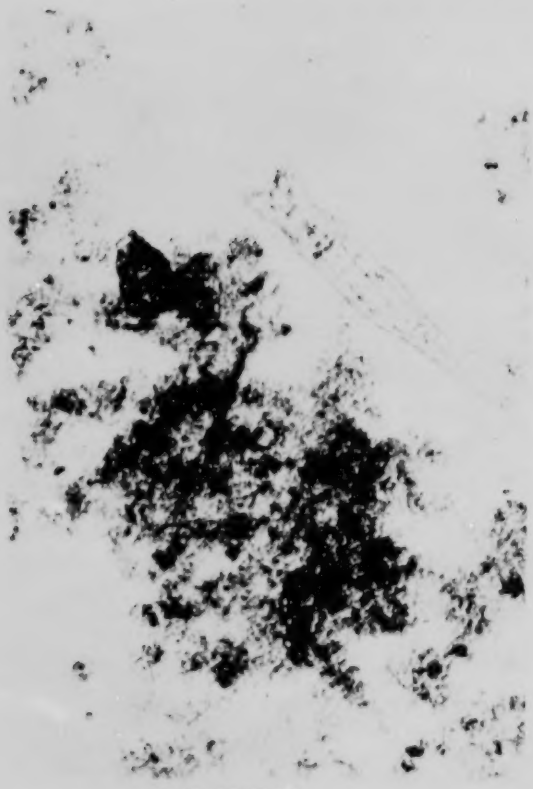
Figure 7-1

HEAD OFFICE: 2005 Drummond Street, Montreal 107, Quebec
BRANCH: Suite 215, Scotia Square, Halifax, N.S.
AGENTS IN ALL PRINCIPAL CITIES

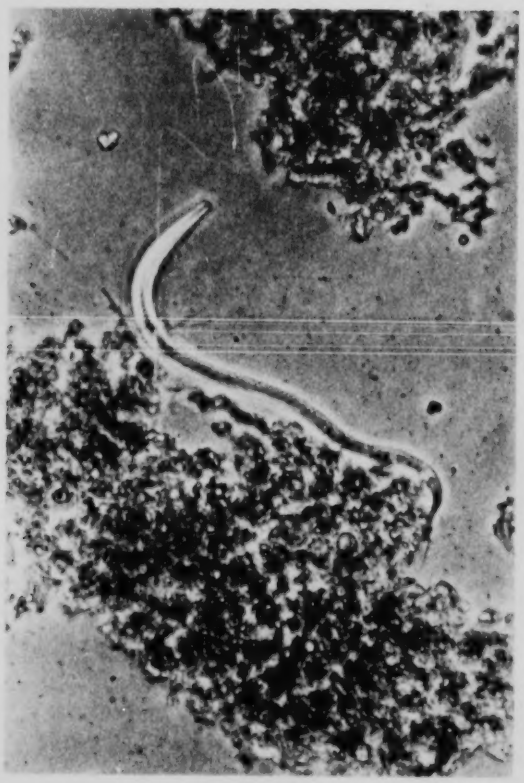
Degremont

Figure 7-2

MICROSCOPIC VIEW OF
ROTIFERS, NEMOTODES AND FIBRE



ROTIFER



NEMOTODE (ROUNDWORM)

FIBRE (WOOD-CLOTH)

Indicating Organisms

Because there is a progressive development of microorganisms in treatment units from the initiation of an activated sludge to a well stabilized condition, the order in which the protozoa appear can be predicted. Varying sludge conditions will affect the order of appearance. Protozoa are therefore classed as indicating organisms. Rotifers on the other hand appear only when the wastewater treatment system is in an advanced state of stabilization. Since they can exist under conditions which are not compatible with the function and survival of the other organisms, the role of rotifers as an indicator organism is suspect. The presence of filamentous bacteria can also indicate, or forewarn of, process control problems.

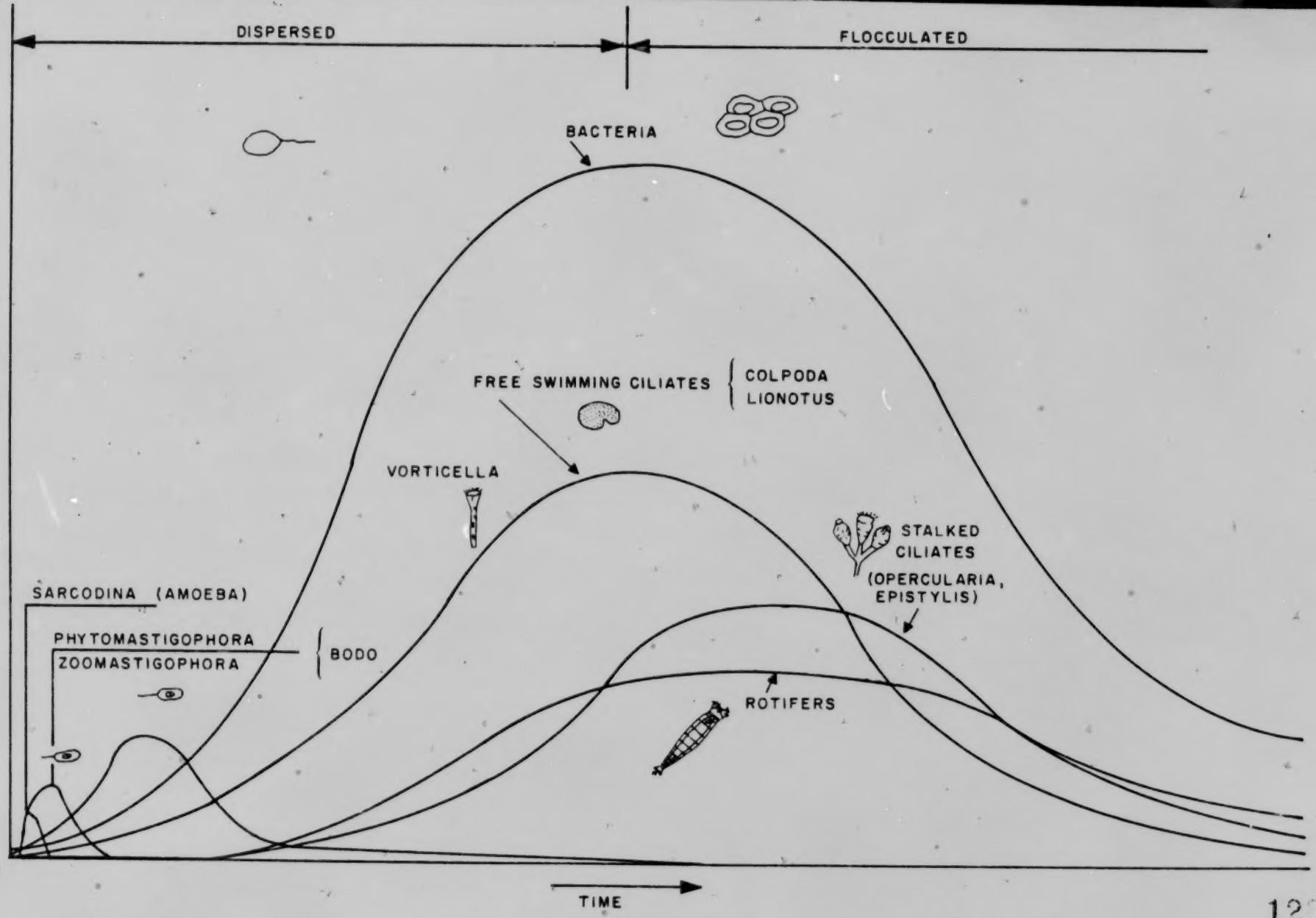
The important aspect from an operation standpoint is the types of protozoa present. *There is a definite succession of protozoa as the sludge is formed and the process becomes efficient.* (See Figure 7-3). Knowing the various protozoa and their significance can be a valuable guide to better activated sludge operations. Protozoa can be observed quickly using a microscope and such observations reflect the immediate condition of the sludge.

By identifying the indicator organisms through regular microscopic examination of the activated sludge, the operator can recognize or even predict problems occurring or likely to occur in the treatment process.

IDENTIFICATION AND EVALUATION

Filamentous Organisms

Filamentous organisms are commonly associated with poor settling or "bulking" sludges, producing sludge volume indexes (SVI) as high as 500. The presence of these organisms is caused by many complex conditions (e.g. organic overloading) and many adversely affect the effluent quality.



7-5

FIG. 7-3 - RELATIVE PREDOMINANCE OF MICROORGANISMS IN ACTIVATED SLUDGE SYSTEMS

In a mixed liquor, there are different types of filamentous organisms which may appear, when viewed at 100X, as a multitude of very fine strands haphazardly interwoven throughout the sludge. See Figure 7-4. Frequently, these filaments are of the *sphaerotilus* species. Also, when "sphaerotilus" is present, the effluent may be clear and sparkling because it does a good job of clarification. However,the sludge settles so badly that mixed liquor is lost over the weirs under normal hydraulic conditions.

Protozoa

Protozoa are single-celled animals which are characterized by the fact that they are microscopic and that they ingest solid food, either alive or previously alive. The types which prevail in any aeration system vary, depending upon the nature of the wastes being treated. Curves in Figure 7-3 show successions of dominant protozoa in relation to the degree of purification. The species of protozoa present in the activated sludge changes with process conditions and effluent quality. Industrial waste systems or systems with a high influx of industrial wastes require careful control to ensure continuous operation at maximum efficiency. Protozoa are more easily affected than bacteria by toxic compounds and other adverse conditions such as oxygen deficiency or excessive organic loads, so regularly observing the changes in protozoa from their normal levels can give an immediate indication of an upset before it has had time to affect the bacteria.

In industrial wastewater treatment plants, the kinds of protozoa may differ from those normally associated with municipal wastewater treatment plants.

For the purpose of the role they play in sewage treatment we will consider three groups of subphylum of protozoa, *Sarcodina, Mastigophora (Flagellata)* and *Ciliophora or Ciliata.* These are considered below in the general order of their appearance in a new aeration system.

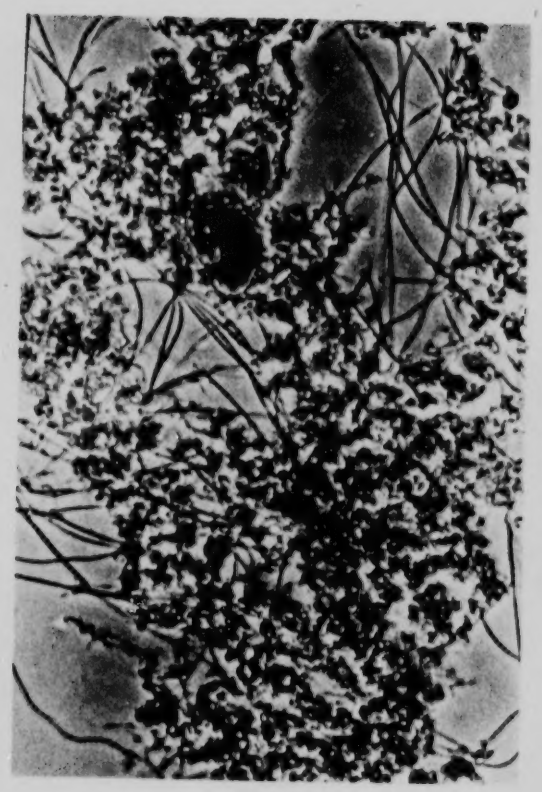


Figure 7-4
MICROSCOPIC VIEWS OF FILAMENTOUS BACTERIA

1. Sarcodina (Figure 7-5)

The distinguishing feature of members of this group is the presence of pseudopodia (false feet). These tiny organisms have an extremely elastic cell membrane and the fluid protoplasm within the cell literally "flows" in the direction that the animal wishes to move. *Protoplasm* is the clear, jelly-like substance in all plant and animal cells. Pseudopodia will also be extended to surround food particles which are then ingested through the cell membrane. The amoeboids are common among this group.

Sarcodina predominate in activated sludge systems during the start-up, or during the system's recovery from a kill.

2. Mastigophora (Flagellata) (Figure 7-5)

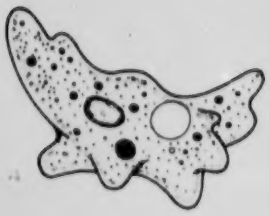
The members of this group are small round or elliptical organisms bearing flagella (tails). These are whip-like appendages which enable the organism to move through the water, usually with a corkscrew motion. At low magnifications (100X), the fast, erratic movement of the tail would not be visible. One or more species of the flagellata may be present; for example, bodo species, monas species.

Mastigophora appear when the bacteria population is low and the food concentration high. As the bacteria increase in numbers, the flagellates cannot compete for the available food and they soon decrease and become insignificant.

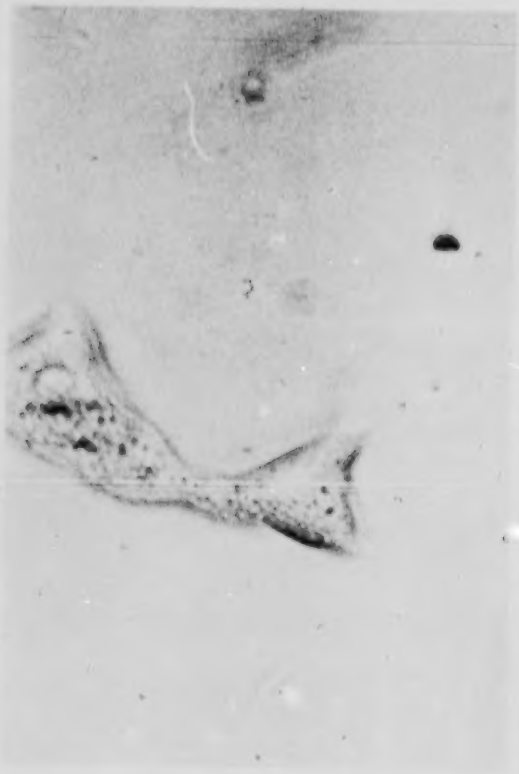
3. Ciliophora or Ciliata

These animals have very fine hairs called "cilia" which cover all or some of their bodies. Cilia are used for locomotion, and the cilia around the gullet are used for capturing food. Possession of cilia is common to all members of this group, although they are more easily seen in some species than in others.

Ciliata are substantially larger than flagellates.



Sketch of Sarcodina
(amoeba)



Microscopic View of Amoeba

Sketch of Mastigophora (Bodo)
(Flaggelates)

Microscopic View of
Mastigophora (Euglena)

Figure 7-5
SARCODINA AND MASTIGOPHORA

Ciliata may be broken down into three groups:

a) The *free-swimming* ciliates, such as paramoecium colpeda and colpidium; (See Figure 7-6)

b) The *crawling* types, of which aspidiscus will be the most common one; (See Figure 7-7)

c) The *stalked* types, which are anchored to a clump of sludge and are either (i) individual; for example, vorticella; or (ii) colonial (grouped); for example, opercularia. (See Figures 7-7 to 7-9)

The presence of ciliophora is an indication of bacterial activity at any given time. The bacterial activity is a measure of the biochemical condition of the system. The ciliates reflect the efficiency of the process with considerable accuracy when observed with the microscope under various day-to-day loading conditions. For example, slug loads of chemical or organic wastes will be indicated by a noticeable reduction of ciliata present. The rise and decline of the types considered normal can indicate an upset some time before the bacteria can be knocked out, because the ciliates are more easily affected by toxicity and load changes.

The free-swimming ciliata are present when there are numerous free-swimming bacteria. The ciliates feed on large numbers of these free-swimming bacteria, reducing the effluent turbidity and BOD to levels found in normal activated sludge systems. Large numbers of free-swimming and crawling ciliates indicate good operation. Flagellates and free-swimming ciliates may indicate low treatment efficiency, whereas the presence of stalked ciliates and some higher forms of life (for example, rotifers) should indicate an efficiently operating system.

Stalked ciliates arise when the free-swimming ciliates are unable to compete for the available food. An activated sludge system which is very stable will have stalked ciliates and usually very few spontaneously moving protozoans.

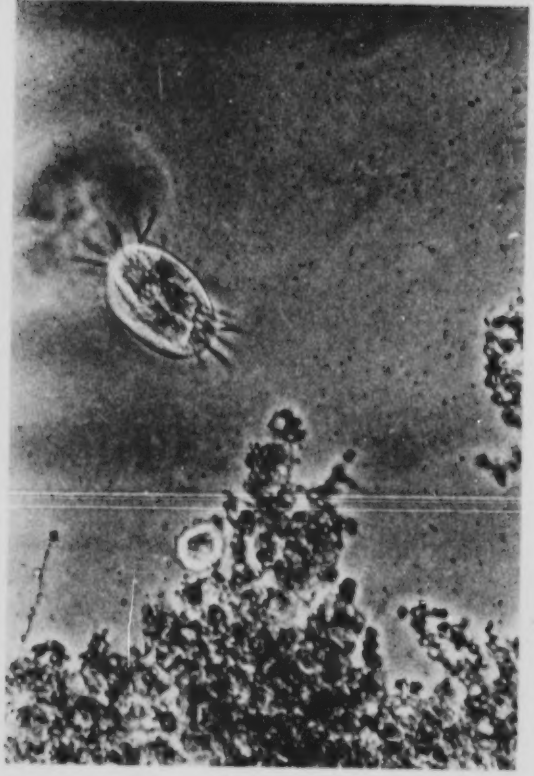
Sketch of Paramecium
(Free-Swimming)

Microscopic View of
Colpidium (Free-Swimming)

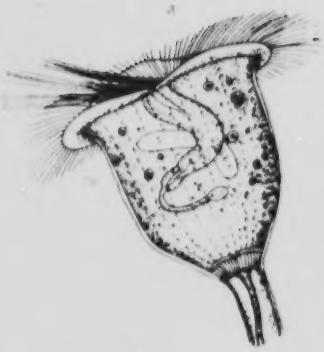
Microscopic View of
Colpoda (Free-Swimming)

Microscopic View of
Lionotus

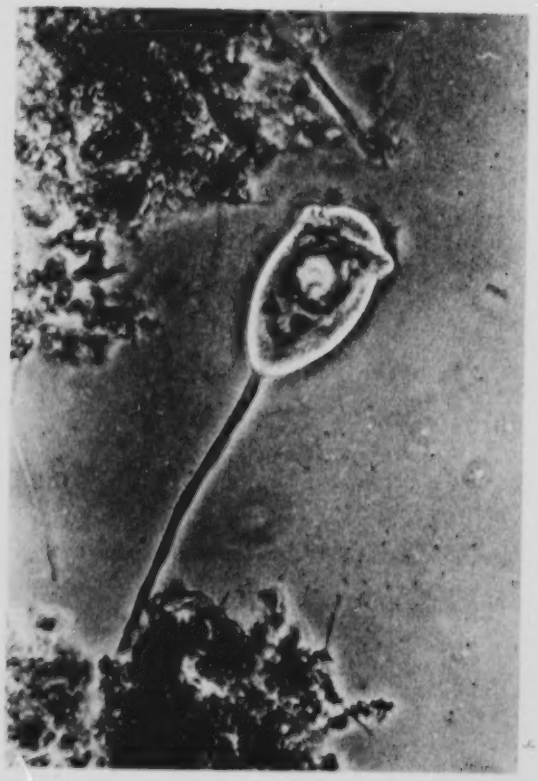
Figure 7-6 CILIATA (FREE-SWIMMING)



Microscopic View of
Aspidiscus (Crawling)



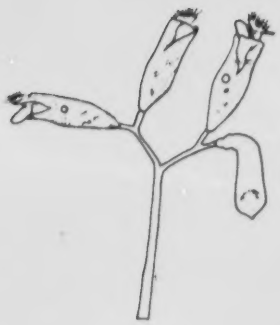
Sketch of Vorticella
(Stalked)



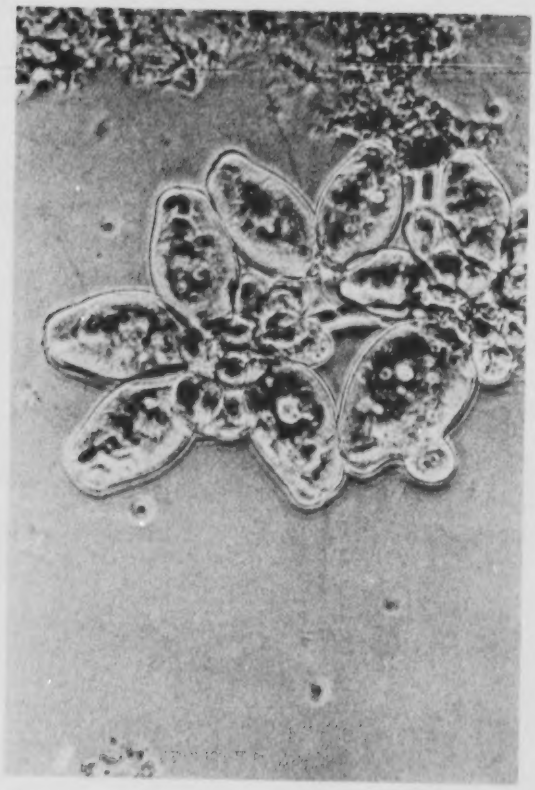
Microscopic View of
Vorticella

Microscopic View of
Vorticella

Figure 7-7 CILIATA (STALKED AND CRAWLING TYPES)

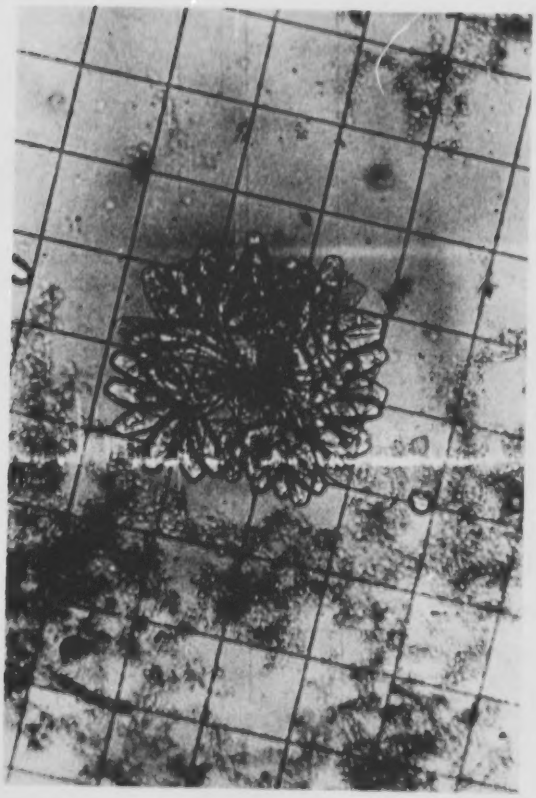


Sketch of Opercularia



Microscopic View of Opercularia

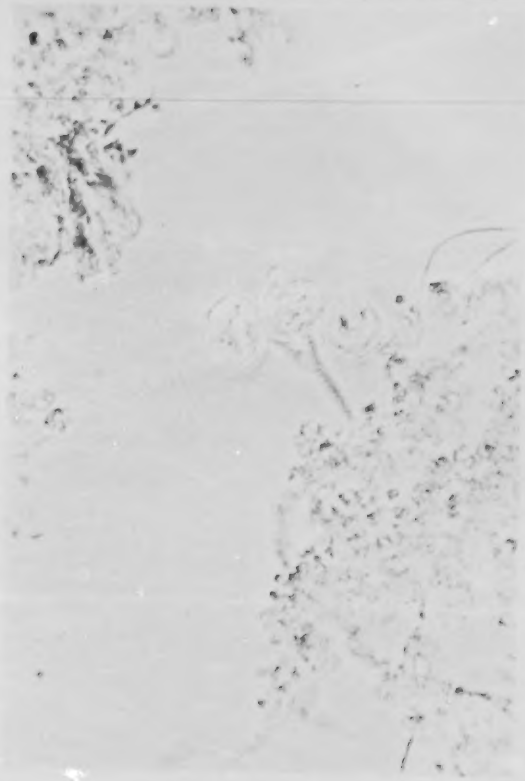
Sketch of Carchesium



Microscopic View of
Large Colony Opercularia

Figure 7-8 CILIATA STALKED TYPES

Sketch of Epistylis



Microscopic View of Epistylos

Figure 7-9 CILIATA STALKED TYPES

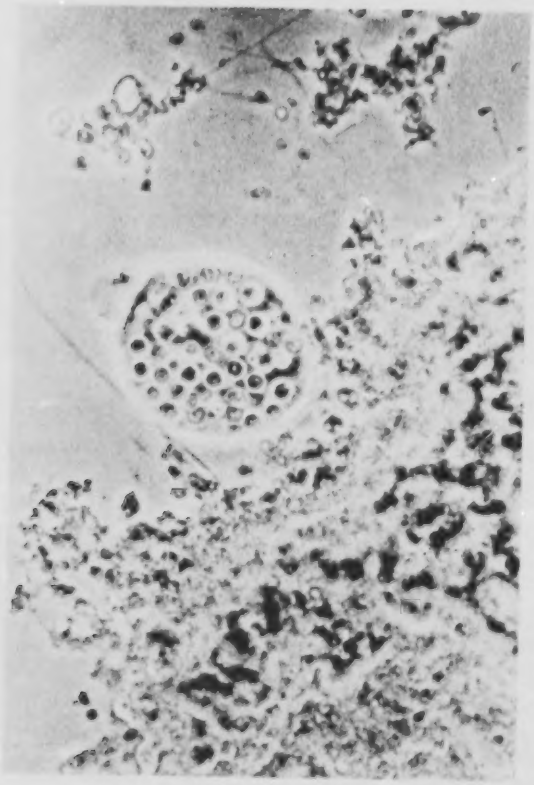


Figure 7-10

Microscopic View of Vorticella Organism Dying

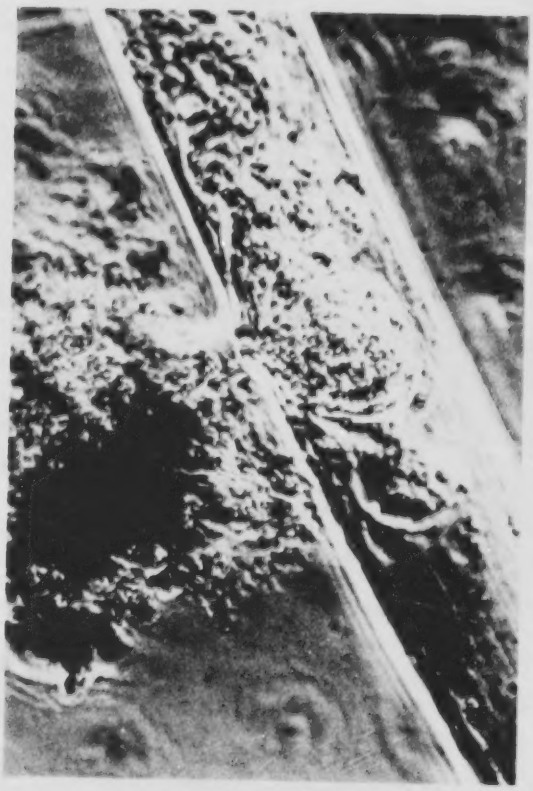


Figure 7-11

Escaping Life Processes

THE MICROSCOPE

Description

Figure 7-12 shows the various parts of the compound microscope. This type of instrument must be used to examine activated sludge. The compound microscope has two separate lens systems. The one nearest the specimen, called the *objective*, magnifies the specimen a certain amount. The *eyepiece*, the second lens system, further magnifies the image produced by the objective. Three and sometimes four different objectives are mounted on the revolving nose-piece so that high and low magnifications may be obtained. The standard objectives may be 10X, 40X, 90X, or on a four objective nose-piece: 10X, 20X, 40X, 90X. In examining protozoa, a 10X eyepiece is combined with a 10X objective to produce an overall magnification of 100X. The higher power objectives may be used for more specific identification.

NOTE: Objectives 10X, 20X, 40X are dry objectives and do not come in contact with the cover slip. The 90X is for oil immersion, that is a drop of immersion oil is placed on the cover slip and the 90X lens is brought into direct contact with the oil.

Most microscopes are pre-aligned by the manufacturer. Except in rare cases, or after misuse, centralization of the optical system should not be necessary.

What to Look for When Buying a Microscope

1. Built-in illumination or an external system which allows variation of light intensity.
2. A condenser system.
3. A movable stage. It is better to have a stage controlled by a coaxial handle rather than a manual push-pull.
4. 10X and 40X objectives.
5. 10X eyepiece.

PARTS OF THE MICROSCOPE

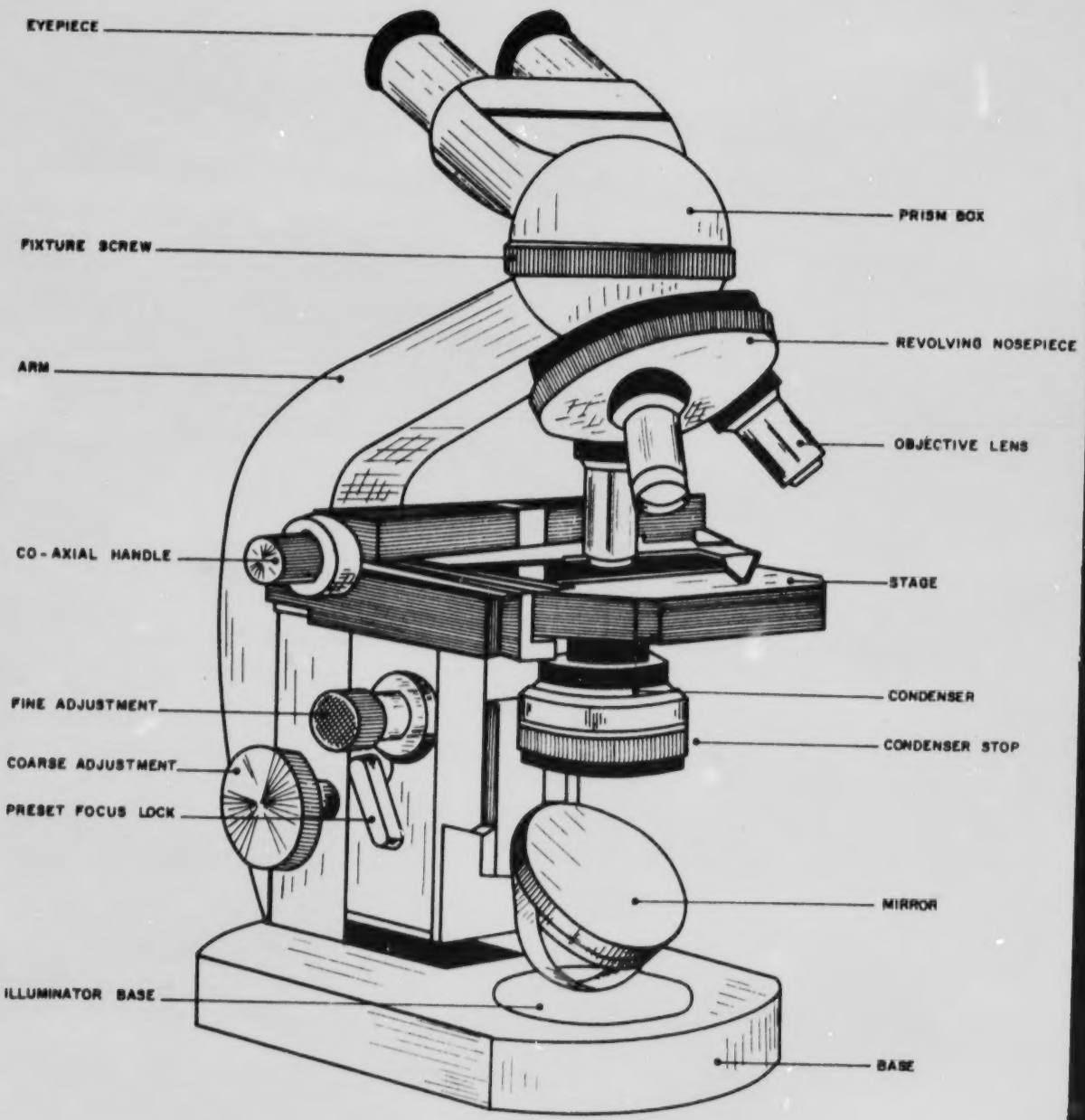


Figure 7-12

Auxiliary equipment should include a light blue filter (daylight type), slides, coverslips and a number of small dropping pipets. A *storage box* or *dust cover* is essential. See Figures 7-13 and 7-14.

The cost of a microscope can vary between $150.00 to $1,500.00 depending upon the individual's requirements in the way of illumination, lenses and auxiliary equipment. A relatively inexpensive instrument is all that is required for the examination of activated sludge.

Maintenance

Simple cleaning of the lens is all that should be undertaken by the operator. This should be external only and *only lens tissue or tissues made for use with glass should be used.* Do not allow dust to accumulate on the instrument. If any moisture gets on any part of it, wipe it off immediately. *Solvents of any kind should never be used.* Adjustments and internal cleaning should be done only by trained microscopists or by the manufacturer.

Use of the Microscope

1. Clean cover slip using "Kimwipes".
2. Use pipet to pick up sludge. Put finger on tip of pipet until other end of pipet reaches bottom of sludge sample. Release finger to allow sludge into pipet. Replace finger on tip of pipet and remove from sample beaker. (Figure 7-15)
3. Allow one drop of sludge from pipet in middle of clear area on glass slide by lifting finger from tip of pipet momentarily, and then replacing finger. (Figure 7-16)
4. Pick up cover slip (cleaned in Step 1) by two corners. Do not put finger on cleaned area.

   Pull cover slip along glass slide towards drop of sludge. (Figures 7-17 and 7-18)

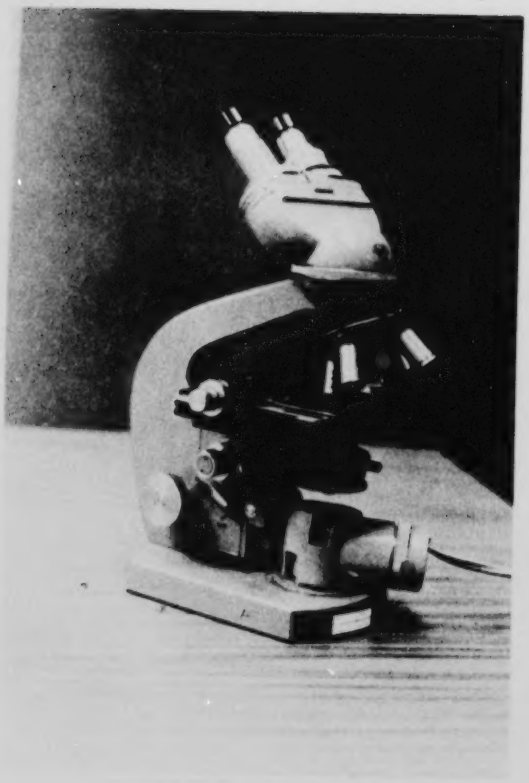


Figure 7-13
Microscope

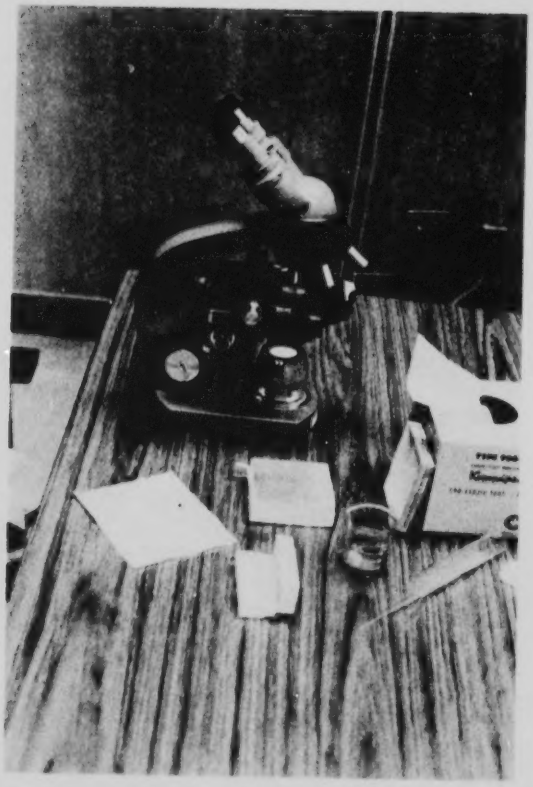


Figure 7-14
Auxiliary Equipment

5. As soon as cover slip touches drop of sludge, allow cover slip to fall onto glass slide. (Figures 7-19)

6. Pick up glass slide. Place on microscopic stage. (Figure 7-20)

7. Move stage up to within approximately 1/8 inch of 10X objective. Look at glass slide through the eyepiece of the microscope. (Figure 7-21)

8. Use the coarse adjustment on the microscope to bring the sludge into the field of focus. (Figure 7-22)

9. Use fine adjustment to refine focus to suit your eyes. Figure 7-23.

10. Determine organisms in the sludge by using the 40X objective. While observing the organism, draw a sketch of what you see.

Note:

It is preferable to use the 10X objective to bring the organisms into focus, *then* change to one of the higher power objectives, the 40X. All that will then be needed is a slight re-adjustment of the fine focus knob. This is because the depth of focus is so small at high power that it is difficult and sometimes almost impossible to use the coarse focussing adjustment; the travel is so great for a small movement of the knob that the slide could be in and out of focus without even having been seen.

If there is too much light, the diaphragm should be adjusted and/or the condenser moved down to provide enough light to properly illuminate the subject matter.

Always keep a sheet of paper and a sharp pencil handy when doing a microscopic examination to keep a record of the types of organisms seen and make sketches of unknown varieties. These may be identified later.

Figure 7-15 Use small pipet to withdraw settled sample from beaker.

Figure 7-16 Transfer one drop of sample to centre of glass slide area.

Figure 7-17

Take clean cover slip. Hold edges. Slide towards drop of sample.

Figure 7-18

Cover slip makes contact with drop of sample. Sample drop begins to spread on glass slide under cover slip.

Figure 7-19

Cover slip is dropped onto glass slide.

Figure 7-20
Secure slide on stage.

Figure 7-21
Using co-axial screws, position slide so that specimen is under objective lens.

Figure 7-22

Using coarse adjustment knob, carefully bring slide in close proximity close to objective lens.

Figure 7-23

Using coarse and fine focus knobs move slide away from objective lens until in focus.

SUBJECT:

ACTIVATED SLUDGE
PROCESS CONTROL

TOPIC: 8

pH MEASUREMENT

OBJECTIVES:

Using a pH Meter (Electrometric Method), trainees will be able to:

1. Standardize the pH Meter.
2. Take the pH reading of a sample.
3. Maintain electrodes in operating condition.

pH

pH is a term that is used to express the level or intensity of the acid or alkaline conditions that prevail in a sample. Technically speaking pH is a measure of the hydrogen ion concentration in solution. Theory shows that in aqueous (= water based) solutions this hydrogen ion concentration ranges from a high of 1.0 mole/l down to 0.000 000 000 000 01 mole/l. Since the International System of Atomic Weights established 1 mole of hydrogen to weigh 1 gram this concentration range could be looked upon as 1.0 g/l to 0.000 000 000 000 01 g/l. Figures of this order are not well suited for practical applications and the Danish technician Sorenson suggested a now universally accepted method of expressing the hydrogen ion concentration using logarithms. The above concentrations are nearly always less than 1 mole/l or 1 g/l and the logarithm would nearly always be negative. Sorenson thus used the symbol p in front of the chemical symbol H (for hydrogen) to change the sign of the logarithmic value to positive and called his scale "pH values". For aqueous solutions the scale ranges from 0 to 14 with 7 being the neutral point where a sample is neither acidic, nor alkaline.

Strong Acid			Weak Acid			Neutral Point			Weak Alkali		Strong Alkali		
0	1	2	3	4	5	6	7	8	9	10	11	12	13	14

pH scale

Measurement of pH is accomplished by means of pH electrodes which develop a potential (voltage) which can be measured by a suitable amplifier (pH meter). In essence these electrodes constitute a cell (battery) whose output potential (voltage) is governed by the pH value of the test solution.

METHODS OF MEASUREMENT

pH Indicators and Indicator Papers

There is on the market a large variety of pH indicators for use with comparators or photometers. Indicator papers for pH are available covering various narrow or wide ranges of pH.

This method of measurement is very useful when an approximate spot check of the pH of a particular waste stream becomes necessary either at a plant or in a sewer system. It must be noted that pH indicators or indicator papers are affected to various extents by the following sample parameters: dissolved solids, colloids, suspended solids, protein, O.R.P. and solvents. If pH readings accurate to 0.1 pH units are desired, the electrometric method should be used.

Electrometric

1. Principle of Operation

   A measurement is made of the millivoltage that is produced, when a sample and a couple of electrodes of a special type are set up as a galvanic cell. The number of millivolts produced depends on the level of acidity in the cell system.

2. Types of Meters

   Several varieties of pH meters are in use. (See Figure 8-1). The basic method for the operation of such instruments is outlined on the following pages.

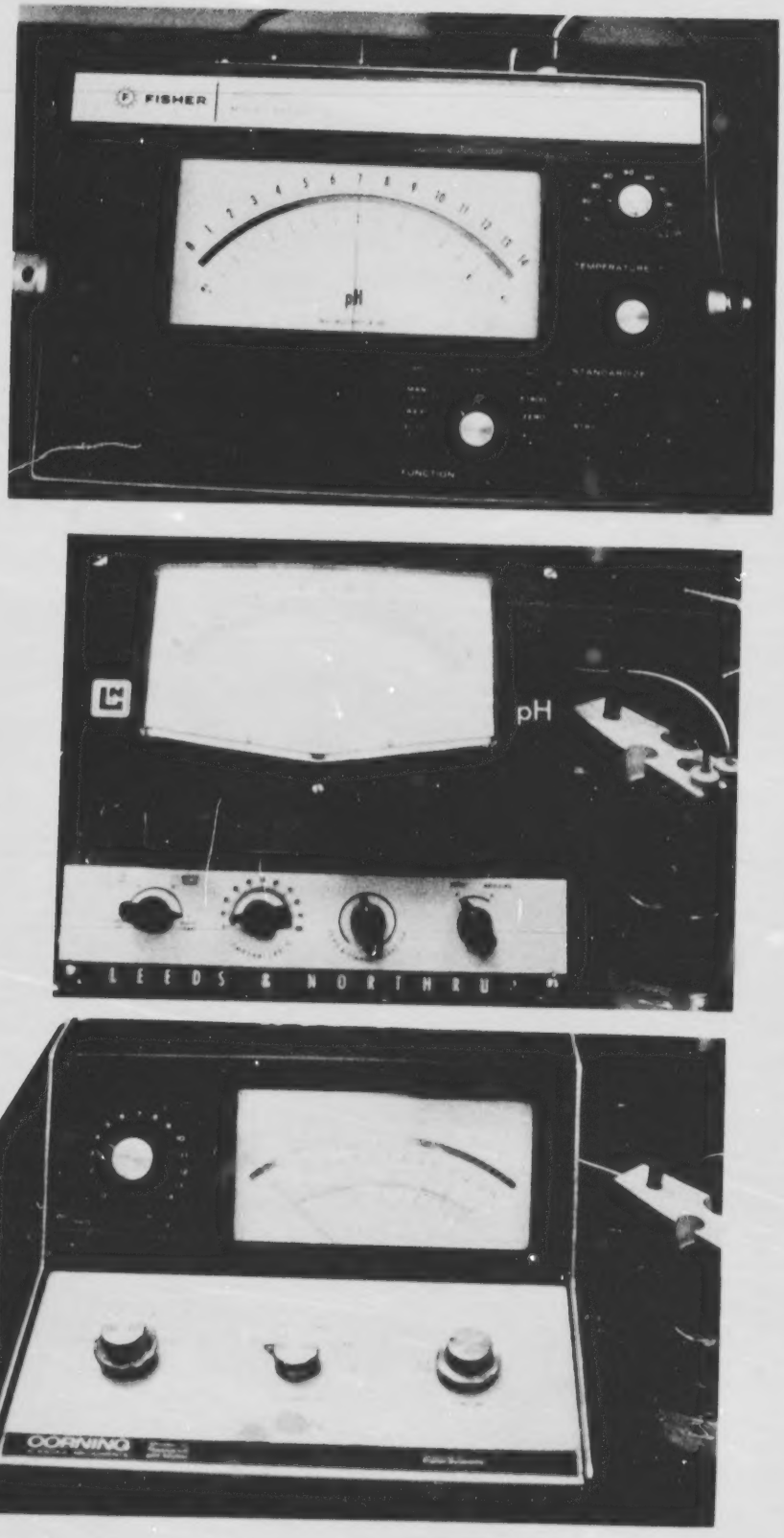


Figure 8-1 pH METERS

Procedure for Standardizing pH Meter

1. With the switch to the "OFF" position, plug the instrument into an A.C. receptacle of correct voltage. If the instrument is battery operated, check the batteries once monthly (or more often if the instrument is used frequently).
2. Plug the electrodes into the jacks provided.
3. If the electrodes are new, place them in distilled water overnight.

   CAUTION: Support electrodes properly, so they do not come in contact with the beaker (use plastic beakers wherever possible).

4. Turn the instrument to "CHECK" or "STANDBY" position and allow to warm up for 3 to 5 minutes.
5. Carefully remove the electrodes from the beaker they were stored in and rinse them with distilled water.

   NOTE: (a) If the reference electrode is fitted with a rubber plug or sleeve, be certain to expose the filler vent hole to the atmosphere by removing the plug or sliding the sleeve away from the hole.

   (b) A drop of distilled water remaining on the tip of the electrodes does not matter.

6. Fill a separate plastic beaker (100-250 ml volume) with buffer solution, pH 7.00.
7. Measure the temperature of the buffer solution and set the temperature compensator to the temperature of the buffer solution.
8. Immerse the electrodes into the beaker, swirl the beaker carefully without touching the electrodes to the beaker sidewalls.

9. Turn the instrument to "READ" and watch the needle: It should deflect quickly towards the pH value of the buffer solution. The response may slow down when the needle is in within 0.2 pH units from the expected reading, but the needle should give a steady reading after about 30 seconds.
10. If the reading does not correspond with the buffer value, adjust the "STANDARDIZATION" or "BUFFER ADJUST" control to position the needle at the correct buffer value.
11. Turn the instrument back to "STANDBY", remove the electrodes from the buffer solution and rinse them off with distilled water.
12. As a quality check, to assure that the meter and probe respond to pH repeat steps 6, 7, 8 and 9, using a buffer solution of a pH value at least 2 pH units away from the first, a buffer 4.00 solution, for example.

    NOTE: Electrodes tend to respond slower at pH values above pH 7.00, so allow 1 minute before taking a final reading if pH is above 7.00.

13. The second buffer reading should be within 0.2 pH units from the expected value. The meter is now calibrated. Turn the instrument back to "STANDBY".
14. Recalibrate the meter daily.

Taking a Reading

1. Repeat steps 5, 6, 7, 8 and 9, using sample instead of buffer.

   NOTE: The temperature compensator must be adjusted to the temperature of the sample and time allowed for the electrodes to reach the same temperature as the sample; for example, if the buffer temperature is 22°C and the sample tempera-

Figure 10-4
Mix sample.

Figure 10-5
Pipet sample.

Figure 10-6
Drain pipet onto filter paper inside brass ring.

ture only 8°C, allow 3-5 minutes for temperature equalization before taking the final reading.

2. Turn the instrument back to "STANDBY", remove the electrodes from the beaker, rinse them well with distilled water and store them in distilled water. Turn the instrument off if it is no longer used.

   NOTE: Solids adhering to the electrodes from samples such as digester samples should be removable by rinsing. Absorbent tissue such as Kimwipes or Kleenex may be used with caution to remove solids that are not removed by rinsing. DO NOT USE PAPER TOWELS!

GENERAL NOTES ON pH METERS AND ELECTRODES

1. Newer solid state (transitorized) pH meters require less warm-up time than the now outmoded tube-type meters. Tube-type meters are best kept turned on in the "STANDBY" position, unless the manufacturer suggests otherwise.
2. The meters are usually sensitive and should not be jarred, for example, when being transported.
3. Although the better meter types are static-proofed, all meters can be affected by static electricity, especially in dry weather. If the needle seems to "stick" in a certain position, breathe on the meter face to put a haze on it. If static electricity was "pinning" the needle, it should now become free. Application of an anti-static agent available as liquid or spray in electronics outlets or better record stores will cure this nuisance problem.
4. Electrodes are available as separate glass and reference electrodes or in single assemblies known as combination electrodes. For pH work,

Figure 10-7

Remove glass fibre disk from Buchner Funnel.

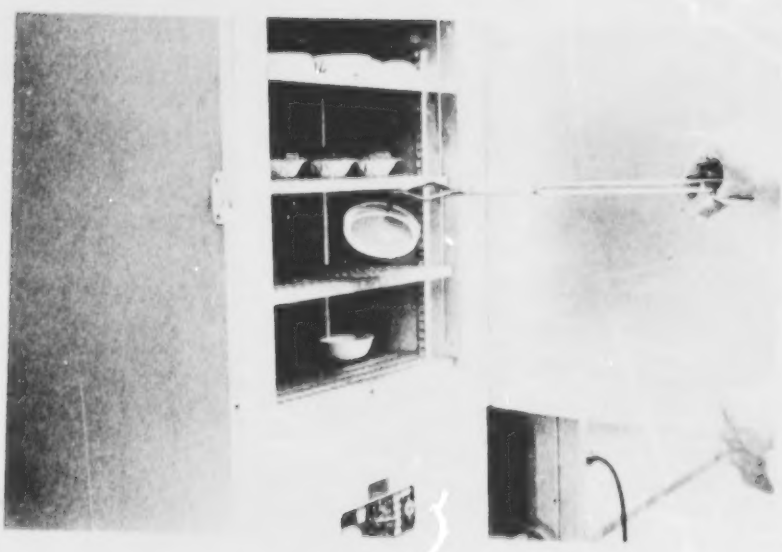


Figure 10-8

Place disk into oven.

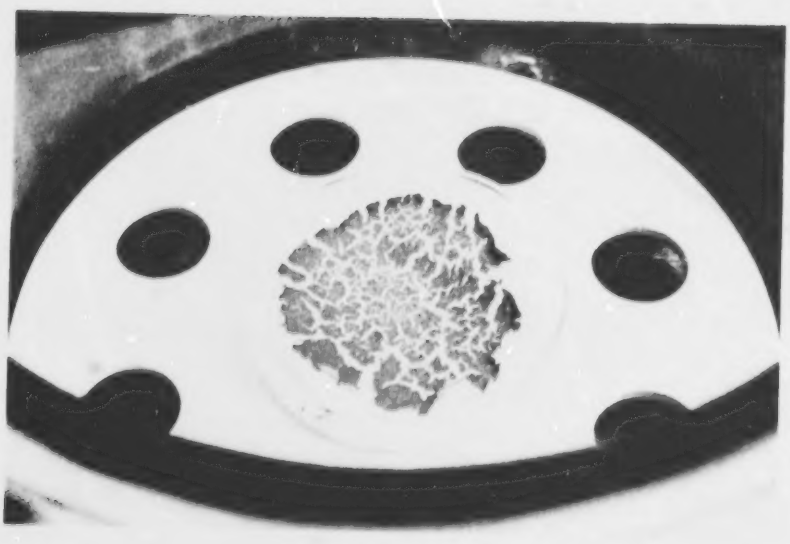


Figure 10-9

Cool disk in desiccator.

both are equally effective, the combination type being more convenient to use since the glass and reference electrodes are packaged into one assembly. Rugged plastic bodied electrodes with protectors for the fragile pH glass bulb are available.

5. The bulb of the glass electrode is constructed of a glass membrane, a few thousandths of an inch in thickness. This makes the bulb extremely fragile and vulnerable. *Scratches on this glass caused by sliding the electrode on the inside wall of a glass beaker can affect pH readings.* When placing the electrode flat on a table, always place the bulb on a soft cloth, sponge, or absorbent tissue.
6. The reference electrodes are normally rugged enough to withstand minor shock and abrasion, but they are also made of relatively thin glass. Keep the electrolyte level high and above the level of the liquid in the beaker. If a pipet or eye-dropper seems inadequate to refill the electrolyte, use a syringe reserved for that purpose. Allowing the electrolyte to dry out can cause permanent electrode failure. A recent development to overcome such problems are sealed gel-filled reference electrodes.
7. After varying periods of usage, the electrodes will become coated with grease from sewage, hardness films from water or final effluents, or caked-on solids from digester samples. NEVER USE HOT WATER TO CLEAN EITHER ELECTRODE. Try immersing the electrodes for a few seconds in a solution having 1 part concentrated hydrochloric acid, and 1 part distilled water to remove film and "caked-on" solids; then rinse well with distilled water. Grease may be removed by wiping the pH electrode bulb with a cloth or tissue soaked with acetone, methyl alcohol or isopropyl

alcohol (rubbing alcohol), then rinsing with lukewarm tap water and replacing in the distilled water beaker. Varsol may be used if none of the other cleaning solutions are available.

8. Buffer solutions can be made up according to "Procedures in Standard Methods", but commercially available buffers, either as a dry salt, concentrate, or ready-to-use solution, are preferable when the quantity used is considered. Buffers should be stored in a dark place, such as in the drawer of a laboratory bench or in a BOD incubator. Exposure to bright sunlight or heat causes rapid decomposition of the buffer by accelerating bacterial and algal growth. Buffers are stable *for a few months (not years)* under optimum conditions, and should be purchased or prepared frequently in small amounts. Some commercially-prepared buffer solutions are labelled with an expiry date and should be discarded after this date.

9. The electrical output of a pH electrode is extremely small: about 0.004 microamperes maximum at room temperatures. Touching the plug or getting it wet may be enough to "short circuit" the electrode. For this reason the contacts on the glass electrode plug should be kept clean by wiping, if necessary, with a clean dry cloth. Tarnished silver contacts should be wiped bright with "Crocus Cloth" or 600 emery or carborundum paper.

10. Failure of the electrodes to produce readings that are within 0.3 to 0.4 of each other in the two buffer standardization techniques previously described, usually signifies defective electrodes.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL TESTS

TOPIC: 9

SETTLING TEST

OBJECTIVES:

The trainee will be able to

1. Demonstrate the settling test.
2. Calculate SVI.

SETTLING TEST

GENERAL

The 30-minute settling test is conducted on mixed liquor to determine the ability of the solids to separate from the liquid in the final clarifier, since the quality of effluent is dependent upon the absence of solids flowing over the effluent weir. The results of the settling test are also used, together with the suspended solids test, to determine sludge volume index. The suspended solids test and settling test should be run on the sample of mixed liquor. This will allow calculation of the Sludge Volume Index (SVI) or the Sludge Density Index (SDI).

The per cent settling rate can be compared for the various days of the week and with other measurements - suspended solids, SVI and per cent sludge solids returned, to provide a record of plant performance and a basis for process control.

APPARATUS

1,000 ml graduated cylinder.

30-minute timer or watch.

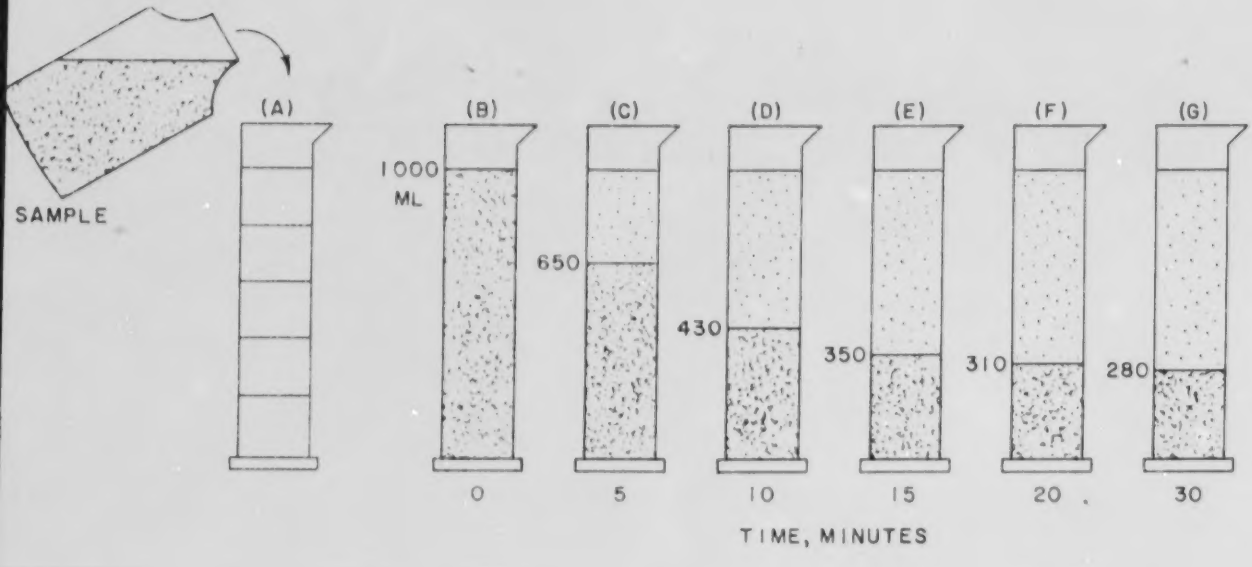


Figure 9-1 Settling of Activated Sludge Solids

PROCEDURE

1. Collect a sample of mixed liquor or return sludge.
2. Gently mix sample and pour into 1,000 ml graduated cylinder. (Vigorous shaking or mixing tends to break up floc and produces slower settling or poorer separation).
3. Record settling solids at 5-minute intervals on a line graph.
4. Calculate % settleable solids - e.g. In Figure 9-1 , % settleable solids after 15 minutes is

$$\frac{350}{1,000} \times 100 = 35\%$$

5. Plot % settleable solids on graph. See Figure 9-2.

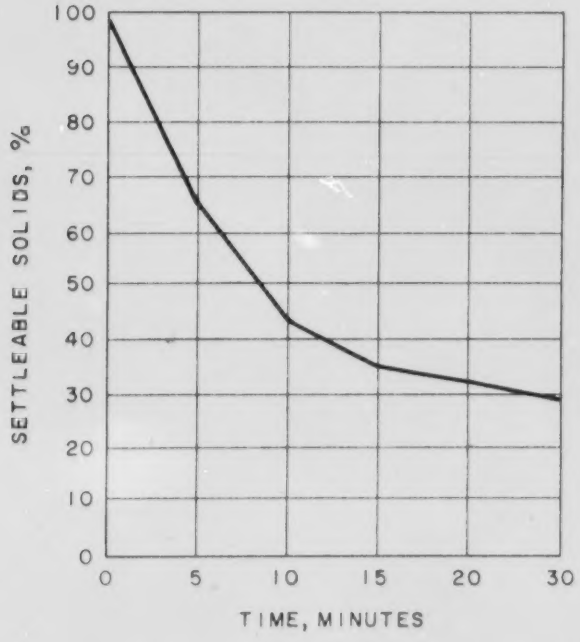
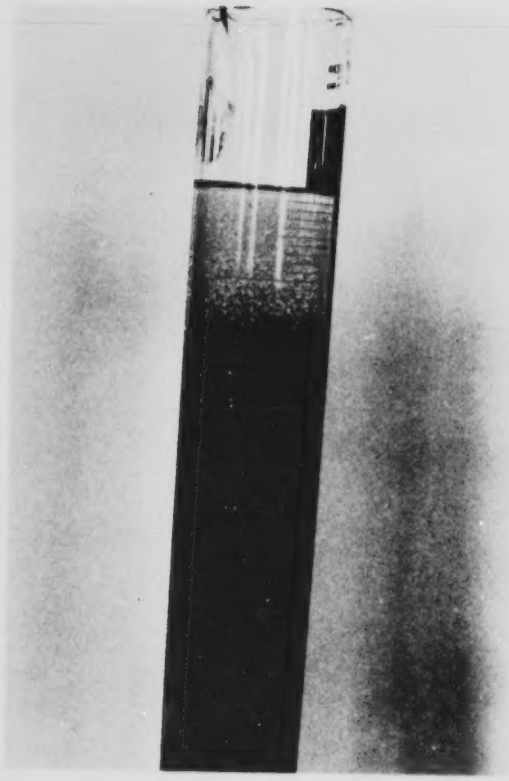


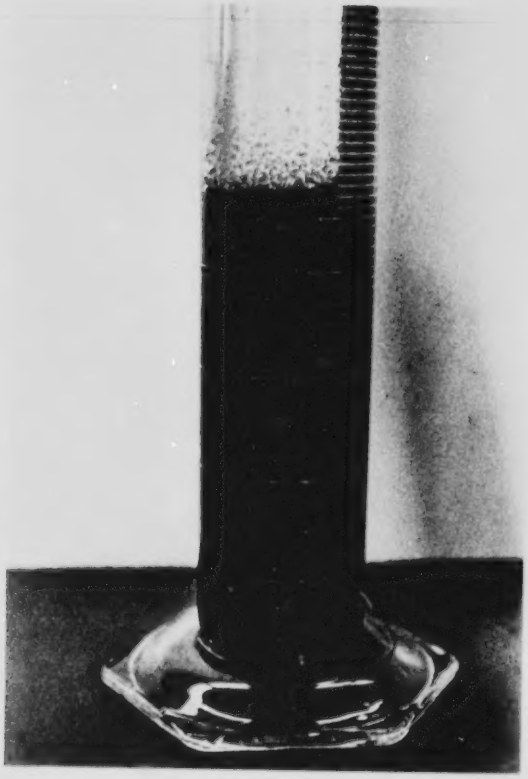
Figure 9-2 Graph Showing Settling of Activated Sludge Solids.

Note: *If a cold sample of mixed liquor containing a high DO level (typically a winter condition) is allowed to warm up significantly during this test, the sludge may rise due to the desolubilization of gases. The test must then be repeated in a location where the sample will not warm up significantly.*

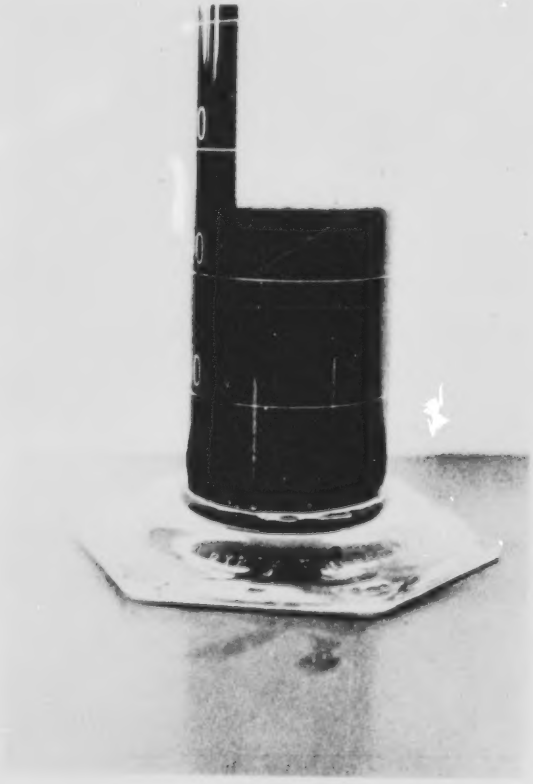
Figure 9-3 SLUDGE SETTLING



After 5 minutes settling.



After 15 minutes settling.



After 30 minutes settling.

SLUDGE VOLUME INDEX (SVI)

The Sludge Volume Index (SVI) is used to indicate the condition of sludge (aeration solids or suspended solids) for settling in a secondary or final clarifier. The SVI is the *volume* in ml occupied by one gram of mixed liquor suspended solids after 30 minutes of settling. It is a useful test to indicate changes in sludge characteristics. The proper SVI range for a plant is determined at the time when the final effluent is in the *best* condition regarding solids and BOD removals and clarity.

Example

Using a 1,000 ml cylinder, after 30 minutes settled solids = 180 ml or 18%.

Mixed liquor suspended solids = 1,500 mg/l.

Calculations

$$\text{Sludge Volume Index (SVI)} = \frac{\% \text{ Settleable Solids} \times 10{,}000}{\text{Mixed Liquor Suspended Solids, mg/l}}$$

$$= \frac{18 \times 10{,}000}{1{,}500}$$

$$= \frac{1{,}800}{15}$$

$$= 120$$

SLUDGE DENSITY INDEX (SDI)

The Sludge Density Index (SDI) is used in a way similar to the SVI to indicate the settleability of a sludge in a secondary clarifier or effluent. The calculation of the SDI requires the same information as the SVI test.

$$SDI = 100/SVI$$

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 10

SUSPENDED AND VOLATILE SUSPENDED SOLIDS

OBJECTIVES:

The trainee will be able to:

1. Carry out the test to determine suspended and volatile suspended solids.

2. Calculate, based on test results,

   a) suspended solids

   b) volatile suspended solids

3. Recall the normal sample aliquot required when testing

   a) raw sewage

   b) primary effluent

   c) mixed liquor

   d) return sludge

   e) final effluent

   f) aerobic and anaerobic digester supernatants

4. Define the following terms:

   a) suspended solids

   b) volatile suspended solids

   c) aliquot

SUSPENDED AND VOLATILE SUSPENDED SOLIDS

GENERAL

The method proposed is based on the 13th edition of Standard Methods. A known sample volume is filtered through a glass fiber filter disk which has been weighed. The disk is then dried and weighed again: an increase in weight is due to the suspended solids in the sample. The disk is ignited at 550°C and weighed once more. The decrease in weight is recorded as volatile suspended solids.

There are several other methods by which suspended and volatile suspended solids may be determined including the Gooch Crucible procedure using either glass fiber disks or an asbestos mat, filter paper methods with low ash papers or Millipore paper, the double-dish method, centrifuge readings, and instrument methods, e.g. optical, radioactive and ultrasonic. However, the method proposed here is fast, easy to perform, and sufficiently accurate.

APPARATUS

1. Glass fiber filter disks, 9 cm diameter
2. Buchner funnel, size 2A for 9 cm filter paper
3. Filter flask or suction apparatus, attachable to vacuum source
4. Brass ring 3½ in. diameter, 1-3/4 in. high
5. Wide mouth pipets, 25 ml, 50 ml and 100 ml
6. Graduated cylinder, 500 or 1,000 ml
7. Convection type drying oven for use at 103°C
8. Muffle furnace for use at 550°C
9. Porcelain dishes to fit inside muffle furnace
10. Desiccator with self-indicating desiccant

11. Analytical balance accurate to 0.1 milligrams
12. Aspirator bulb
13. Forceps, flat nose, bent tip
14. Whatman No. 3 filter paper, 9 cm
15. Petri dishes, 100 mm diameter, 15 mm high
16. Wash bottle, polyethylene squeeze type
17. Long tongs
18. Pipet tray or stand

NOTE: If suspended and volatile suspended solids are only determined periodically (once or twice per week), the convection type drying oven should be preheated overnight, or at least one-half day prior to the solids determination. If suspended and volatile suspended solids are determined continuously, the convection type drying oven should be operated continuously at 103°C. The muffle furnace should be preheated to 550°C one or two hours before use.

FILTER DISK PREPARATION

1. Place glass fiber disk in desiccator overnight.
2. The following day, use forceps to pick up the filter disk; place on the analytical balance and weigh. (Figure 10-1)
3. Record the weight as "DRIED WEIGHT".

ANALYSIS OF SAMPLE(S)

1. Place a Whatman No. 3 filter paper in the Buchner funnel. Place the prepared glass filter disk with its wrinkled surface facing *upward* on the filter paper. (Figure 10-2)
2. Connect the above-assembled filtration apparatus to a vacuum source. Turn on the vacuum source.

3. Using distilled water, wet the filter disks to ensure that they are properly seated. (Figure 10-3)

4. Pick up brass ring. Gently lower ring into Buchner funnel and place it concentric with filter disks.

5. Using a clean wide-mouth pipet with an aspirator bulb on the upper end or a graduated cylinder, measure out the selected volume of well-mixed sample. Let pipet drain by itself until liquid stops dropping by itself onto filter paper inside brass ring. The vacuum source will now "pull" the sample through the filter disk. Wash disk and brass ring with distilled water and allow to drain; disconnect the vacuum source. (Figures 10-4, 10-5 and 10-6)

6. Remove the brass ring without touching the residue on the filter disk. Using the forceps, grasp the outer edge of the disk (left clear and without any residue by use of the brass ring). Transfer the disk to its previously labelled Petri dish. Using tongs, pick up the dish and place in the convection type drying oven for one hour, with the temperature at 103°C. Remove and cool in desiccator. (Figures 10-7, 10-8 and 10-9)

7. Using forceps, pick up filter disk, place on analytical balance and weigh. (Figure 10-10)

8. Record the weight as "DRIED WEIGHT + SOLIDS".

9. If <u>volatile suspended solids</u> are to be determined, do the following:

   a) Use forceps to pick up filter disk whose "dried weight" and "dried weight + solids" have been determined, and place on procelain dish. (Figure 10-11)

   b) Use long tongs to pick up dish and transfer to muffle furnace which has been preheated to 550°C. (Figure 10-12)

...cont. pg. 10-9

Figure 10-1

Pre-weigh glass filter disk.

Figure 10-2

Place disk in funnel.

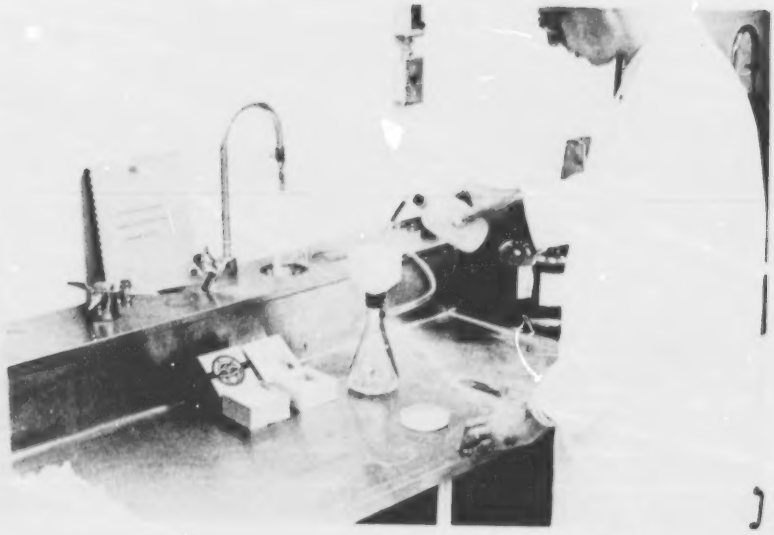


Figure 10-3

Wet filter disk using distilled water.

Figure 10-10
Weigh disk.

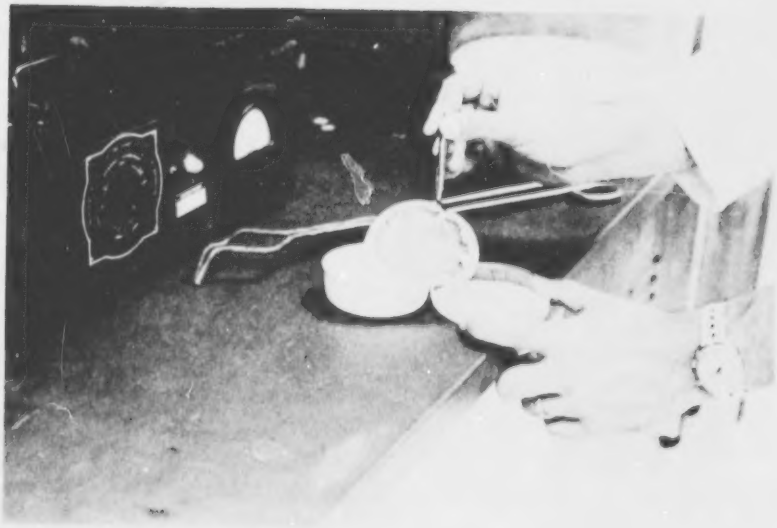


Figure 10-11
Transfer disk to porcelain dish.

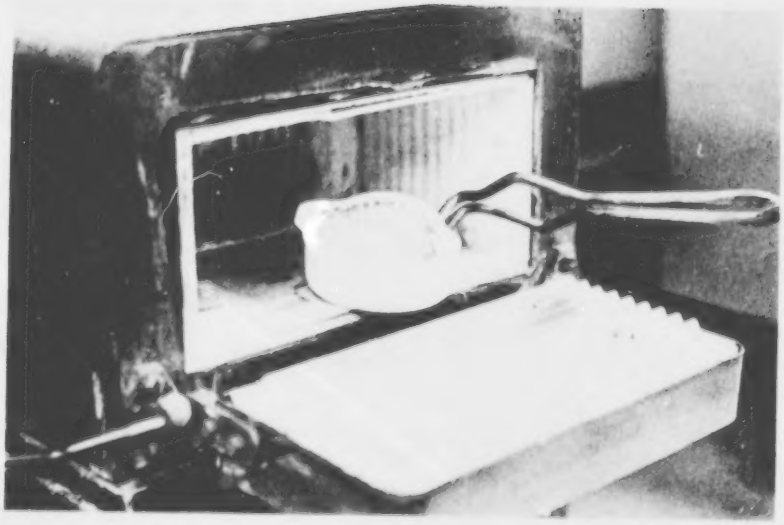


Figure 10-12
Place disk in muffle furnace.

Figure 10-13 Removed cooled disk from desiccator.

Figure 10-14 Weigh ignited disk.

c) Ignite filter disk with its suspended matter for 15 minutes at 550°C. Again using long tongs, pick up porcelain dish; transfer dish to desiccator and allow to cool for 5 minutes before totally replacing desiccator lid. Allow to cool to room temperature. (Figure 10-13)

d) Using forceps, pick up filter disk, place on analytical balance and weigh. Record this weight as the "ASHED WEIGHT". (Figure 10-14)

NOTE: The volatile suspended solide consists of the weight loss on ignition.

CALCULATION

The suspended solids and volatile suspended solids can now be determined using the following formulae:

1. To determine SUSPENDED SOLIDS (mg/l)

$$SS = \frac{(B-A) \times 1000 \times 1000}{V}$$

where SS = Suspended solids (milligrams per litre)
A = Dried Weight (grams)
B = Dried Weight + Solids (grams)
V = Volume of sample used for analysis (millilitres)

EXAMPLE

Sample volume of mixed liquor used = 50 ml.
Dried disc weight (tare weight) = 0.0615 gm.
Dried weight of disc and solids = 0.2145 gm.

Calculate the MLSS in mg/l
NB. 1 gm = 1000 mg
Applying this data to the formulae for suspended solids calculation:

$$MLSS = \frac{(B-A) \times 1000}{V} \times 1000$$

where MLSS = suspended solids mg/l
A = dried disc weight (tare weight in grams)
B = dried weight of disc and solids in grams
V = volume of sample used in ml

$$\text{thus MLSS} = \frac{(214.5-61.5) \times 1000}{50} \text{ mg/l}$$

$$= \frac{153 \times 1000}{50} \text{ mg/l}$$

$$= 3060 \text{ mg/l}$$

2. To determine VOLATILE SUSPENDED SOLIDS (mg/l)

$$SS = \frac{(B-C) \times 1000 \times 1000}{V}$$

where SS = Suspended solids (milligrams per litre)
B = Dried Weight of disc and solids (grams)
C = Ashed Weight, after burning (grams)
V = Volume of sample used for analysis (millilitres)

EXAMPLE

Sample volume of mixed liquor used = 50 ml.
Dried weight of disc and solids = .2145 gm.
Ashed weight, after burning = .2099 gm.

Calculate the volatile suspended solids (mg/l)

$$VSS = \frac{(B-C) \times 1000 \times 1000}{V}$$

$$= \frac{(.2145 \text{ gm} - .2099 \text{ gm}) \times 1000 \times 1000}{50 \text{ ml}}$$

$$= \frac{(.0046) \times 1000 \times 1000}{50} = 92 \text{ mg/l}$$

SELECTION OF PROPER SAMPLE ALIQUOT

1. Raw Sewage

Normally 50 or 100 ml aliquots are sufficient. If raw sewage is *low* in suspended solids due to run-off, wet weather conditions, or in filtration, larger sample volumes (250 ml) may be required. Non-homogenous samples may have to be blended.

2. Primary Effluent

Normally 50 or 100 ml aliquots are sufficient. Clear-looking effluents, such as that obtained from chemical treatment, may require aliquots of 200 ml for the analysis.

3. Mixed Liquor

Normally 50 ml aliquot is sufficient. Filamentous sludges may be difficult to filter; in exceptional cases, the sample aliquot may have to be reduced to 25 ml.

4. Return Sludge

Normally 25 or 50 ml are sufficient. Filtration may be affected by the quantity of filamentous growth present: the more filamentous growth, the harder the filtration.

5. Final Effluent

Normally one litre is required for crystal clear effluents. If suspended solids are easily visible, 200 or 500 ml are sufficient. During plant upsets where effluent quality has deteriorated, 100 ml or 50 ml are sufficient.

6. Digester Supernatants

Anaerobic digester supernatant varies considerably, and with it, the ease of filterability. Normally, 50,25, or 10 ml are sufficient.

Aerobic digester supernatant is usually very clear. Normally 200-500 ml are sufficient. However, should the supernatant quality deteriorate, the sample aliquot may have to be reduced to 100, 50 or even 25 mls.

7. Digester Sludges

Because of the high solids concentrations found in digester sludges, a Total Solids Determination is recommended.

TABLE 10-1 SELECTION OF PROPER SAMPLE ALIQUOTS

Sampling Point	Aliquot Required (normal)	Aliquot Required (abnormal)
(a) Raw Sewage	50 or 100 ml	250 ml
(b) Primary Effluent	50 or 100 ml	200 ml
(c) Mixed Liquor	50 ml	25 ml
(d) Return Sludge	50 ml	25 ml
(e) Final Effluent	One (1) litre	200 or 500 ml
" "	" " "	100 or 50 ml
(f) Digester Supernatants		
Anaerobic digester	50, 25 or 10 ml	--
Aerobic digester	200-500 ml	100, 50 or 25 ml
(g) Digester Sludges	Total Solids Determination	--

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 11

TOTAL SOLIDS AND TOTAL VOLATILE SOLIDS TESTS

OBJECTIVES:

The trainee will be able to

1. Carry out the test to determine total solids and total volatile solids.
2. Calculate, based on test results,
   a) Total Solids
   b) Total Volatile Solids
3. Recall:
   a) The purpose of the Total Solids Test.
   b) On what it is normally carried out.
4. Define:
   a) Total Solids
   b) Volatile Solids

TOTAL SOLIDS AND VOLATILE SOLIDS TEST

GENERAL

Total solids measure the combined amount of suspended and dissolved matter in a sample. The amount is determined by weighing the residue after completely evaporating the liquid portion of a measured sample. The total solids are comprised of volatile solids (mainly organic matter of animal or plant origin), and fixed solids (mainly inorganic compounds such as mineral salts, sand and silt). Volatile solids are those solids which are lost after ignition at 550°C.

Total solids are normally used as a measure of the concentration of sludges which are difficult to filter, since the dissolved solids form an insignificant fraction of the total solids present. This test is normally done on raw and digested sludges.

APPARATUS

1. Porcelain evaporating dishes, capacity 35-50 ml or aluminum foil dishes if total solids only are desired.
2. Balance, capable of weighing at least to the nearest 0.01 gm, or trip balance and weights.
3. Drying oven at 103°C or infra red heat lamps.
4. Muffle furnace
5. Desiccator
6. Asbestos Mats

PROCEDURE FOR TOTAL SOLIDS

1. Dry the evaporating dish by ignition in a muffle furnace at 550°C for half an hour. Take the dish out of the furnace, cool for a few minutes on an asbestos mat, then put the dish in a desiccator to cool to room temperature. Slide the cover so

that the desiccator is slightly open when the dish is hot. Close completely only when the dish is nearly cooled. *Why? Leaving a hot dish in a sealed desiccator can make it impossible to remove the cover when the desiccator and contents have cooled. If foil dishes are being used, dry in the oven, not the muffle furnace.*

2. Weigh the dish to the nearest 0.01 gm and record weight as *weight of empty dish.*

3. Pour in about 25 ml of well mixed sludge sample and weigh the dish plus wet sample to the nearest 0.01 gram. *Record the weight of dish and wet sample.*

4. (a) Put the dish and sample in the oven and dry overnight. If there is no fume hood or canopy, it is best to put the sample in the oven just before going home.

OR

(b) Under a canopy, place the dish and sample under a heat lamp placed about 4-6 inches away. Evaporate the sample until it is almost dry, then put it in the oven for an hour.

5. Cool the dish with the dried sample in a desiccator.

6. Weigh the dried residue in the dish to the nearest 0.01 gram and record *weight of dish and dried sample.*

CALCULATION OF TOTAL SOLIDS

$$\% \text{ Total Solids} = \frac{\text{Wt. of dish \& dried sample - Wt. of empty dish}}{\text{Wt. of dish \& wet sample - Wt. of empty dish}} \times 100\%$$

PROCEDURE FOR TOTAL VOLATILE SOLIDS

1. Use the dried sample obtained in determining total solids.

2. Preheat the muffle furnace to 550°C.

3. Using tongs set the dish just inside the furnace.

4. With the door open, allow the sample to burn slowly (with no flame).

5. After the sample is burned, set the dish in the furnace and close the door.

6. After five minutes or so, open the door in order to introduce oxygen and to check the sample to see if it is completely burned.

7. Close the door, and leave the dish in the furnace for about another half hour and check again. If the residue or dish has a black carbon deposit, leave it in the furnace for an extra 15 minutes and check again.

8. When there is no evidence of carbon in the ash or on the dish, remove the dish from the furnace using tongs.

9. Cool for a few minutes on an asbestos mat.

10. While still using tongs, place the dish in the desiccator, leaving the cover slightly open until the dish (and desiccator) return to about room temperature. Close the desiccator, cool to room temperature.

11. Weigh the dish plus ash to the nearest 0.01 gm and record *weight of dish and ash.*

CALCULATION OF TOTAL VOLATILE SOLIDS

% Total Volatile Solids =

$$\frac{\text{Wt. of dish \& dried sample - Wt. of dish \& ash}}{\text{Wt. of dish \& dried sample - Wt. of empty dish}} \times 100\%$$

Note: Weight of dish and dried sample and weight of empty dish were previously recorded when determining Total Solids.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL TESTS

TOPIC: 12

DISSOLVED OXYGEN ANALYSIS

OBJECTIVES:

Operator will be able to:

1. Name and describe three methods for the analysis of DO.
2. List the advantages and disadvantages of using
   a. The DO Meter
   b. The Hach DO Kit
3. Demonstrate the method of DO analysis using a DO meter.
4. Determine DO using the Winkler Method.

DO ANALYSIS

METHODS OF ANALYSIS

The dissolved oxygen level of a sample or contents of a basin or tank can be determined by a membrane type DO meter or the chemical, modified WINKLER METHOD.

DO METERS

General

Measurement of the dissolved oxygen (DO) concentration using a DO Meter, which are now common in many plants, is a good substitute for the Sodium Azide Modification of the Winkler Method. *Use of a DO Meter offers the following advantages:*

1. DO Meters are fast and accurate.
2. Results on DO Meters are unaffected by the floc present in the water.
3. Some DO Meters give direct readings in milligrams per litre (mg/l) or parts per million (ppm) while others give direct readings in per cent saturation. Simply take the meter to the site, put the probe into the liquid to be measured, turn on the meter, allow the meter to stabilize about 1 minute, and read the scale.
4. A direct reading of the liquid temperature is possible when using some DO meters.

The disadvantages are:

1. Some gaseous reactive compounds, such as sulphides, can interfere by fouling the probe.
2. The membrane area must be cleaned after each use by rinsing with water.
3. The batteries must be kept charged for use of the meter at any time.
4. Recognition of probe failure may be difficult when caused by poisoning or puncture.

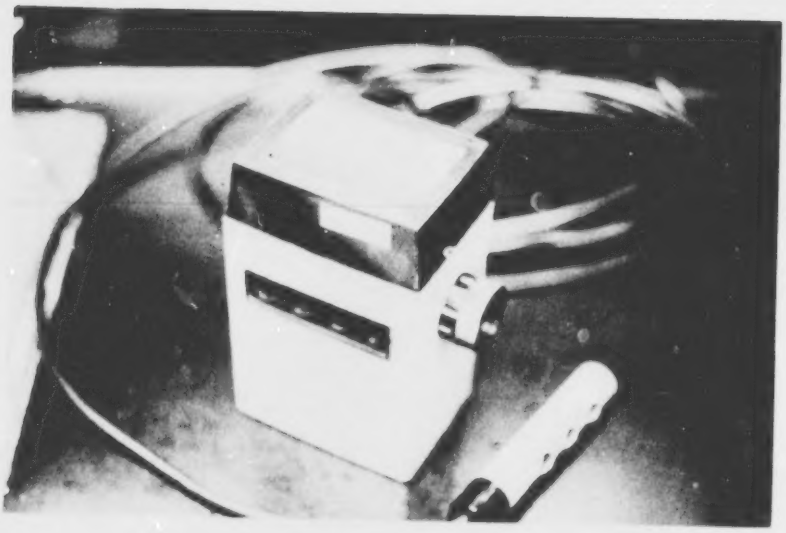


Figure 12-1 DO METER AND PROBE

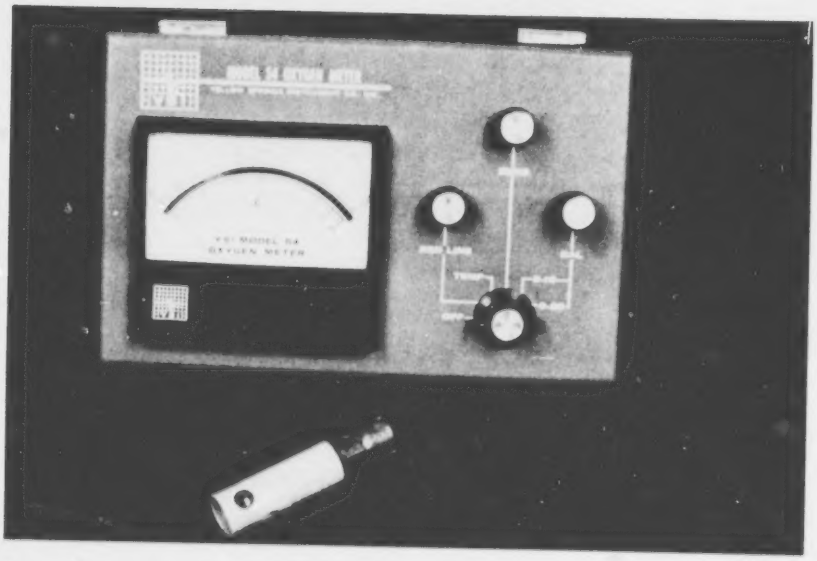


Figure 12-2 DO METER AND PROBE

CALIBRATION OF THE DO METER AND PROBE

Calibration should be carried out regularly if the DO Meter is to be maintained according to the Manufacturer's Maintenance Manual accompanying the meter. *Calibration should be done at least every 2 weeks.*

Zero Checking the DO Probe

It is advisable to check the response of the probe in a solution that contains zero DO. All membrane type DO probes become sluggish in response at DO levels of less than about 1 mg/l or 10% O_2 saturation. Some probes develop a "memory" of some low level of DO and will not readily read values less than this "memory" which can be as high as 1 mg/l.

To carry out a zero check, proceed as follows:

1. Prepare a zero DO standard solution by making up a 5% solution of sodium sulphite (Na_2SO_3) and adding a small crystal of cobalt chloride ($CoCl_2$).
2. Immerse the probe into this solution, turn the meter to read D.O., and watch the response of the meter movement. USUALLY the reading rises briefly, then drops sharply, slowing down as the needle approaches zero.
3. When the needle has stopped moving, or after 3 to 5 minutes, expose the probe to a high level of DO (e.g. air or aerated water).
4. Again, immerse the probe into the sodium sulphite solution and observe the meter response.
5. This cycling of the probe from zero DO to high DO should be repeated until the meter will easily read zero in the sodium sulphite solution.

Zero readings may not always be attainable especially with older DO sensors. Readings of 0.1 to 0.2 mg/l are acceptable in that case. Probes that do not approach a zero reading after repeated cyclings may require storing in the sodium sulphite solution overnight.

The "zero" adjust position on most DO meters is designed to electronically adjust the circuit to a simulated zero DO meter reading.

Do not use this adjustment to set the meter reading to zero when the probe is immersed in sodium sulphite solution and the meter switch set to the "read" position.

Calibration - Laboratory Procedure

After the zero check has been carried out, calibration of the meter is carried out as follows:

1. Take a sample (preferably tap water) which does not contain substances that interfere with either the probe reading or the modified Winkler Method.
2. Divide the sample into two even portions (A&B).
3. Using modified Winkler Method, measure the DO in portion (A) of the sample.
4. Using the DO Meter and probe, measure the DO in portion (B) of the sample.
5. Compare the DO values obtained in (3) and (4).
6. If the results coincide, the DO meter is calibrated.
7. If the results do not coincide, adjust the meter according to Manufacturer's specifications until it agrees with the modified Winkler Method.

Alternative Procedure for Calibration

A convenient and sufficiently accurate alternative method of calibrating the DO meter and probe is outlined for those not having access to the apparatus required to perform Winkler DO determinations.

It is sometimes difficult to perform steps (3) and (4) of the above method without altering the dissolved oxygen content by accidental aeration of one of the portions. Thus, the following alternative method of calibration may be used:

1. Take approximately 750 ml of distilled water, demineralized water, or tap water that is not excessively high in dissolved solids. Transfer this water to a clean 1 litre stoppered Erlenmeyer flask, or clean stoppered tall cylinder. The water should be at approximately room temperature.

2. Shake this water vigorously for 2 minutes to entrain as much air as possible and cause the sample to become saturated with oxygen.

3. Once saturated, this sample will keep its oxygen saturation level for some period of time unless it becomes contaminated or its temperature is allowed to change drastically.

4. Following is a list* of dissolved oxygen concentrations in mg/l attainable by air saturating a sample of water at various temperatures:

^{o}C	^{o}F	AIR SATURATED WATER DO mg/l
17	62.6	9.7
18	64.4	9.5
19	66.2	9.4
20	68.0	9.2
21	69.8	9.0
22	71.6	8.8
23	73.4	8.7
24	75.2	8.5
25	77.0	8.4
26	78.8	8.2
27	80.6	8.1

*Standard Method, 13th ed., page 480-1.

5. Immerse the DO probe which has been rinsed with tap water into the vessel containing the air saturated water, making sure that all the membrane surface and temperature sensing elements are also immersed.

6. If the probe is not fitted with a means of mechanical agitation, provide the necessary agitation by raising and lowering the probe about one inch two to three times per second, without exposing the membrane surface to the air.

   Note: The probe will not respond properly unless the sample is moving past the membrane at about 0.5 ft/sec or faster. This velocity is provided by the above means of agitation.

7. Measure the temperature of the water used for calibration and set the temperature compensator dial on the meter to the measured value. Many meters have automatic temperature compensators, which eliminate the need for this adjustment.

8. Select the proper air saturation value from the above table and while agitating the probe adjust the calibration control on the meter to the selected proper DO value.

   Note: If the meter scale is calibrated in percent oxygen saturation, rather than mg/l or ppm DO, set the meter to 100% on the scale.

9. The meter is now calibrated and ready for use. When taking measurements on a sample stream, be certain to provide the necessary velocity of sample past the probe. For example, aeration sections normally provide sufficient sample velocity, whereas final clarifiers would not.

   Note: When not in use, always keep the membrane in the tip of the probe from drying by inserting in a flask of water.

   WHY? If allowed to dry out, the probe can lose its accuracy until it is re-conditioned.

DO Sample Preservation of Mixed Liquor Samples

The DO present in the aeration tank (mixed liquor) is being depleted by the continuous activity of the micro-organisms. To prevent this and to ensure that a correct reading is obtained, 10 ml of copper sulphate-sulphamic acid reagent for each litre of sample should be added (as a preservative) to the sample container before taking the sample. This chemical preservative will kill the micro-organisms. The copper sulphate reacts with the alkalinity in the sample, forming a copper hydroxide floc which, as it settles, helps to remove the particles of sludge in the sample. The sulphamic acid, besides killing the bacteria, acts to prevent nitrite oxidation.

To obtain and preserve a sample for DO analysis by the Winkler or Hach Method:

1. Add 10 ml of copper sulphate-sulphamic acid reagent for each litre of sample to be collected in the sampling bottle.
2. Use a *tall* bottle having a capacity of about 1 quart. An assembly such as the one shown in Figure 12-3 can be used to fill the bottle, ensuring minimum liquid-air contact.
3. Lower the sampling device into the aeration tank in such a way that it will fill without entraining air.
4. Stopper the bottle and mix well by turning it upside down 4 or 5 times.
5. Allow the solids to settle until there is 50% or more clean liquor above the sludge.
6. Siphon the clear liquor into a BOD bottle. Keep the end of the siphon tube at the bottom of the receiving bottle to avoid aerating the sample, and fill until the liquor overflows the bottle. Stopper the bottle.

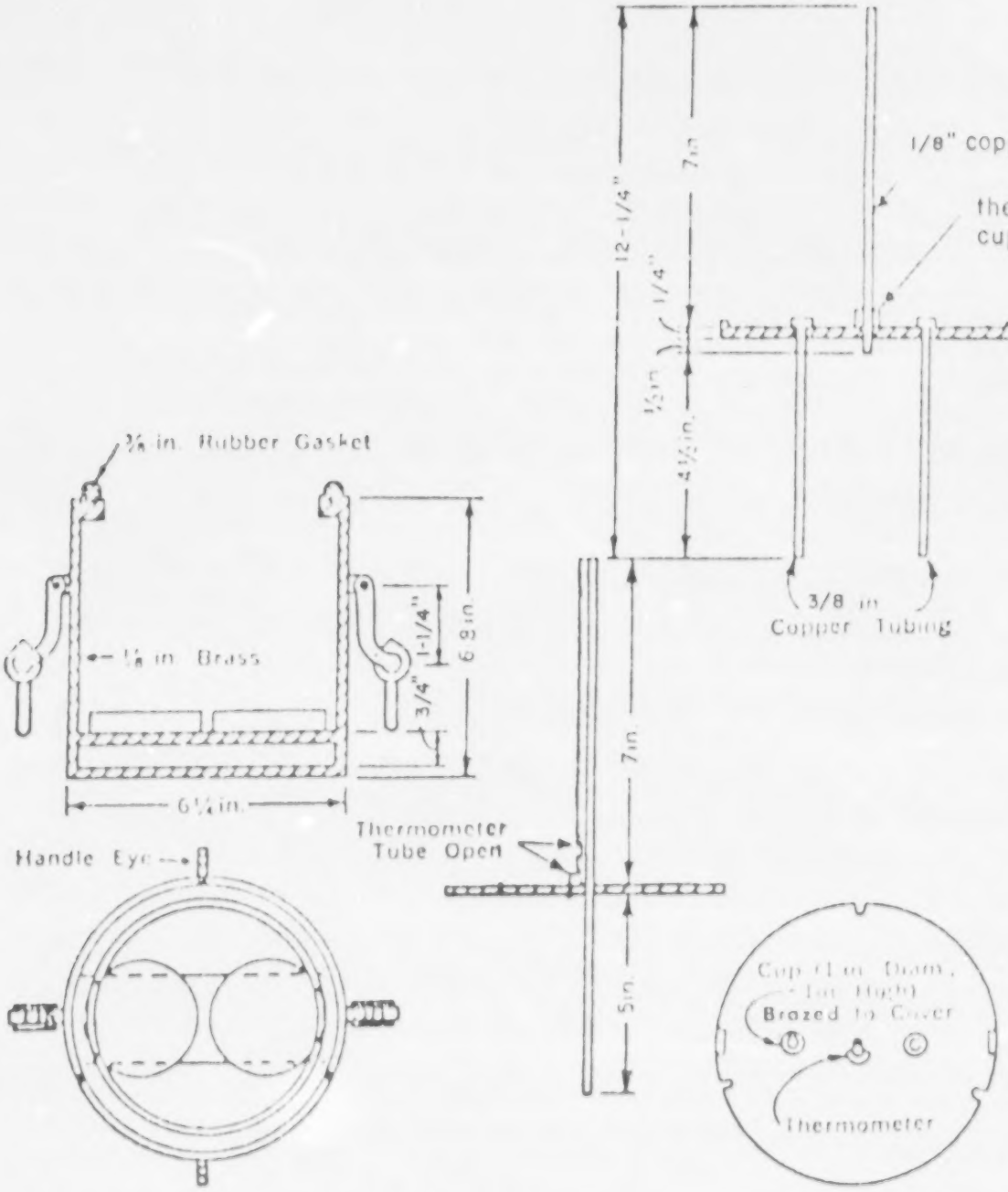


FIGURE 12-3 DO AND BOD SAMPLER ASSEMBLY

DO NOT CREATE TURBULENCE WHEN TRANSFERRING LIQUOR INTO THE BOD BOTTLE. THIS LEADS TO AIR ENTRAINMENT.

WINKLER METHOD

The principle of the Winkler Method is that it releases iodine chemically in proportion to the amount of dissolved oxygen originally present in the sample. Using a standard solution of sodium thiosulphate as a titrant, the amount of iodine and consequently the amount of oxygen present can be determined. It should be noted that the only solution which must be accurately made up (or standardized) is the sodium thiosulphate solution. Preparation of reagents is outlined further on in this topic.

Apparatus Required

1. Buret, capacity 10, 25 or 50 ml, preferably with a Teflon stopcock.
2. Buret support and stand.
3. BOD bottles.
4. Erlenmeyer Flask (200 or 250 ml capacity).
5. Three 10 ml pipets (Serological Pipets) or three 2 ml automatic pipets.
6. One 100 ml graduated cylinder.

Procedure (Figures 12-4 to 12-18)

1. Remove the stopper, and add 2 ml of manganous sulphate solution with the tip of the pipet *slightly below* the surface of the liquid.
2. Using a fresh pipet, add 2 ml of alkaline iodide azide solution, with the tip of the pipet *slightly below* the surface of the liquid.

NOTE: SAFETY PRECAUTIONS

(i) When transferring solutions, samples etc., by pipette from one bottle to another, use a rubber bulb or other automatic pipette, NOT THE MOUTH.

(ii) Use safety glasses for shielding against possible splashing.

(iii) Wear protective clothing (such as a lab coat, for example) to prevent damage to clothing.

3. Make sure that no air bubbles are trapped by tapping the side of the bottle near the shoulder with the stopper. Replace the stopper.

4. Mix thoroughly by turning upside down several times. Do this over a sink since there will be liquid in the seal around the stopper.

5. Allow to settle about half-way. Mix again by inverting and allow to settle a second time. (After addition of the manganous sulphate solution and the alkaline-iodide-azide solution, the samples can be left, if necessary, to complete other work.)

NOTE: When the alkaline-azide-iodide reagent is added, a dense precipitate will form. If this precipitate is pure white, there is no oxygen present; if the precipitate is amber-coloured, there is oxygen present.

6. Remove the stopper and add 2 ml of concentrated sulphuric acid above the liquid surface, letting the acid run down inside the neck of the bottle. Use another pipet for this reagent.

7. Replace stopper and mix by inverting. Do this over a sink. The amber solution of free iodine produced is not stable and should be titrated immediately.

8. Measure 100 ml of the solution in a 100 ml graduated cylinder. Transfer it to an Erlenmeyer Flask.

9. Fill the buret to the zero mark with 0.025N sodium thiosulphate.

10. Add sodium thiosulphate from the buret at a fast rate (steady stream) while mixing the contents of the flask by swirling. Slow down the rate of titrant addition as a pale yellow colour is reached.

11. When the solution is pale yellow, add about ½ ml of starch solution (an eyedropper-full). The sample will turn blue and the titration is continued, adding the sodium thiosulphate dropwise until the solution is colourless. This is the END POINT.

    NOTE: If the colour does not appear when the starch indicator is added, the titration has been carried too far and must be repeated. If the solution turns brownish black, the starch indicator has been added too early and the procedure must be repeated.

12. Record the amount of sodium thiosulphate solution used in ml.

13. When disposing of the sample (both the titrated portion and that remaining in the BOD bottle), leave the water running in the sink to thoroughly flush the acid down the drain.

Calculation

The concentration of dissolved oxygen in the original sample is equal to *twice* the number of ml of sodium thiosulphate used when 100 ml of solution are titrated.

e.g. The titration required 2.3 ml of sodium thiosulphate solution; therefore, the dissolved oxygen concentration was 2 x 2.3 = 4.6 mg/l.

THE HACH DO KIT

This method is essentially a Winkler dissolved oxygen determination with all chemicals supplied in kit form.

1. It is a small, completely self-contained kit.
2. It is easy to transport.
3. It is easy to use (see procedure).
4. It is a modification of the Sodium Azide modification of the Winkler Test.
5. The Hach DO Kit is suited for plant applications. All the chemicals are contained in small plastic pillows, with the exception of the titrating solution. This keeps them uncontaminated and they will last a long period of time. The titrating solution, which is phenylarsene oxide, is very stable. By avoiding contamination, and keeping it well stoppered, it will remain stable for an exceptionally long period of time.

NOTE: FOLLOW THE SAMPLING AND ANALYTICAL INSTRUCTIONS CAREFULLY TO PRODUCE RESULTS COMPARABLE TO THE WINKLER METHOD.

Procedure Using the Hach DO Kit

1. Fill the DO sample bottle with the water to be tested by allowing the water to overflow the bottle for 2 or 3 volumes. Be certain there are no air bubbles in the bottle.
2. Add the contents of one Dissolved Oxygen I Powder Pillow (Manganous Sulphate) and one Dissolved Oxygen II Powder Pillow (Alkaline Iodide-Azide). Stopper in a manner to keep out air. Shake to mix and allow the floc that is formed to settle half-way in the bottle.

3. Remove the stopper and add the contents of one Dissolved Oxygen III Powder Pillow (Dry Acid). Re-stopper and shake to mix. The floc will dissolve and a yellow colour will develop if oxygen is present. *This is the prepared sample.*

4. Fill the sample measuring tube level full with prepared sample and pour it into the 1 oz. titrating vial.

5. Using the dropper provided, add PAO Solution, swirling to mix, and counting each drop. The titrating end point is reached when the sample has turned colourless. Use starch indicator if available to obtain sharper end point. The mg/l Dissolved Oxygen is equal to the number of drops used.

NOTE: It is a bit tricky to stopper the DO sample bottle without getting an air bubble trapped in the bottle. To avoid the air bubble, tip the sample bottle slightly, and insert the stopper with a quick thrust. This will force the air bubbles out.

All the above directions are supplied with the kits and include a procedure for Low Range Dissolved Oxygen where each drop of PAO is equal to 0.2 mg/l.

PREPARATION OF REAGENTS

Copper Sulphate - Sulphamic Acid

1. Dissolved *approximately* 32 gm of technical grade sulphamic acid (NH_3SO_2OH) without heating in about 475 ml of tap or distilled water.

2. Dissolve 50 gm of technical grade copper sulphate crystals ($CuSO_4.5H_2O$) in about 400 ml of tap water.

3. Mix the two solutions.

4. Add *approximately* 25 ml of concentrated acetic acid.

Manganous Sulphate Solution

1. Weigh out about 480 gm of manganous sulphate ($MnSO_4.4H_2O$).

2. Dissolve in 400 to 600 ml of distilled water.

3. Add distilled water to make up volume to 1 litre.

NOTE: Filtration of the solution as recommended in "Standard Methods" may be necessary if there is an oily film on top, or if there is evidence of insoluble black matter.

The solution, using reagent grade manganous sulphate, is a clear pink colour.

Alkaline Iodide-Azide Solution

1. Weigh about 500 gm of sodium hydroxide (or about 700 gm of potassium hydroxide).

2. Dissolve the sodium hydroxide in 500 to 600 ml of distilled water in a Pyrex beaker, cooling the solution in a water or ice bath.

3. Weigh about 150 gm of potassium iodide (KI) (or 135 gm of sodium iodide, NaI).

4. Dissolve the potassium iodide in about 200 to 300 ml of distilled water. When the sodium hydroxide solution is cool, add the potassium iodide solution to it slowly while mixing.

5. Dissolve about 10 gm of sodium azide (NaN_3) in 50 ml of distilled water. *CAUTION: Sodium azide is unstable and in acidic solution could be explosive and also releases toxic fumes.*

6. Add the sodium azide solution to the cooled alkaline iodide solution.

7. Dilute with distilled water to 1 litre.

Sulphuric Acid

Use concentrated reagent grade sulphuric acid. Handle it carefully because it is a strong acid. It will "burn" holes in cloth, especially cotton, and can cause severe irritation of the skin.

NOTE: ALWAYS ADD ACID TO WATER - NEVER WATER TO ACID.

Starch "Solution"

1. Make a thin paste of 3 gm of starch (soluble, potato, arrowroot, etc.) with a small amount of distilled water.
2. Pour into 500 ml of boiling distilled water.
3. Add 1.25 gm/l of salicylic acid for preservation.
4. Allow to cool and settle overnight.
5. Decant, saving the clear supernatant.

Sodium Thiosulphate Solution (0.025N)

NOTE: 0.025N Sodium Thiosulphate Solution may be purchased from some chemical supply houses, or prepared as follows:

1. Weigh out, as accurately as possible, 6.20 gm of sodium thiosulphate crystals ($Na_2S_2O_3.5H_2O$). Do *not* dry before weighing.
2. Dissolve in distilled water and make up to 1.00 litre with distilled water.
3. Add about 5 ml chloroform or about 0.4 grams of sodium hydroxide pellets as a preservative.

   NOTE: If this solution is to be used for plant dissolved oxygen tests only, it will be sufficiently accurate. However, if it is to be used in BOD determinations, it should be standardized against 0.025N Potassium Dichromate (Reagent g). Because the sodium thiosulphate solution is unstable,

it should be replaced every month or standardized weekly if it is used for BOD's.

Potassium Dichromate Solution (0.025N)

NOTE: 0.025N Potassium Dichromate Solution may be purchased from some chemical supply houses, or prepared as follows:

1. Put about 5 gm of reagent grade potassium dichromate ($K_2Cr_2O_7$) in a 50 ml beaker or a weighing bottle.
2. Dry the potassium dichromate at 103°C overnight in the oven.
3. Cool in desiccator.
4. Weigh out exactly 1.226 gm, transferring it to a 1 litre volumetric flask.
5. Dissolve in distilled water; make up to the 1 litre mark.
6. Transfer the solution to a reagent bottle for storage. No solutions should be stored in volumetric flasks.
7. This solution is stable and will not have to be replaced unless it becomes contaminated or is allowed to evaporate significantly.

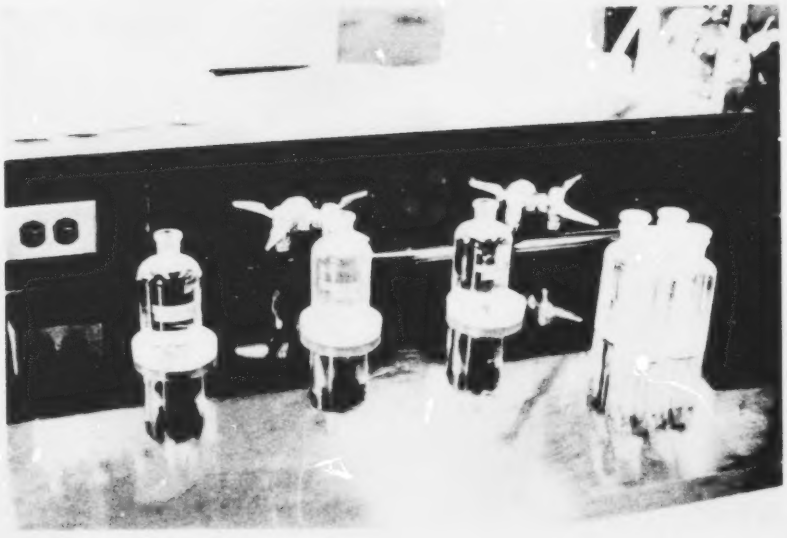


Figure 12-4 Reagents and DO samples.

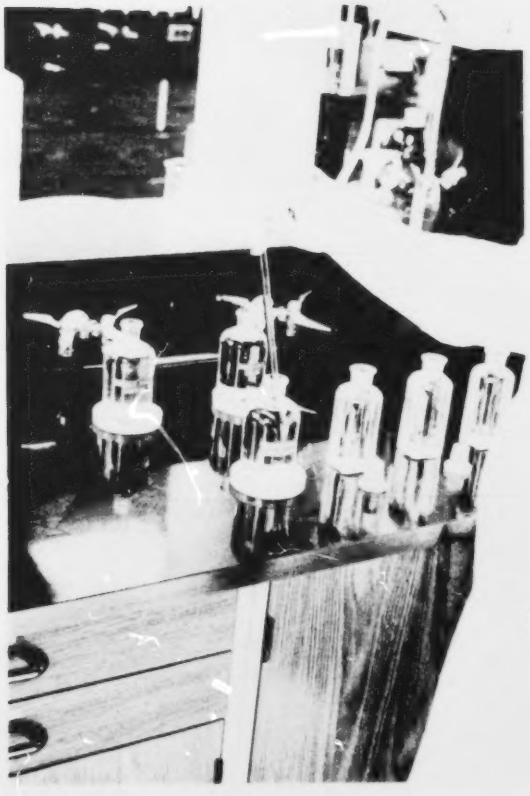


Figure 12-5 Pipetting of magnanous sulphate reagent.

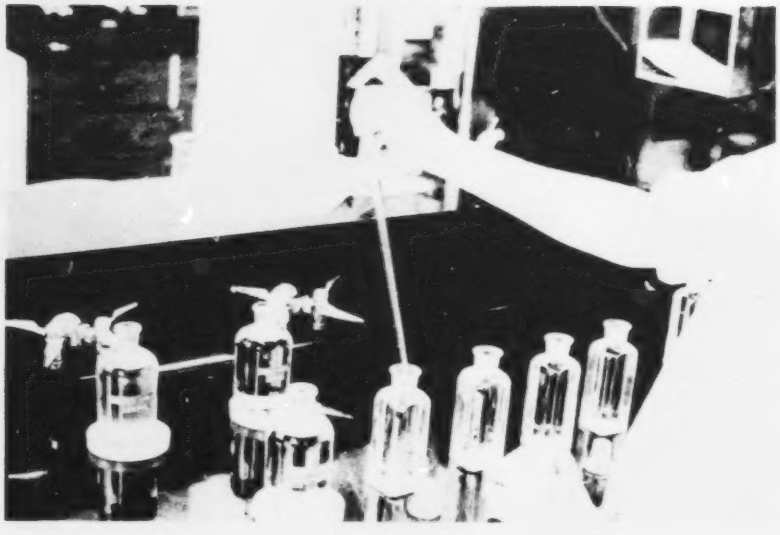


Figure 12-6 Addition of manganous sulphate reagent.

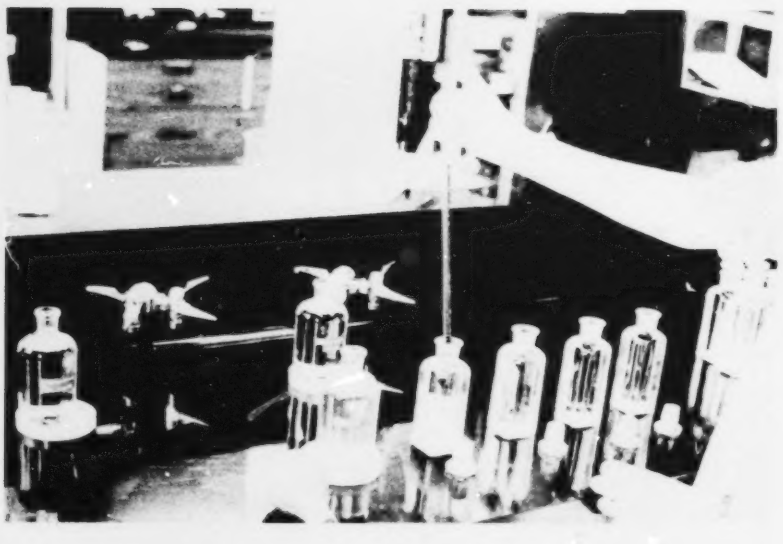


Figure 12-7 Addition of alkaline-azide reagent.

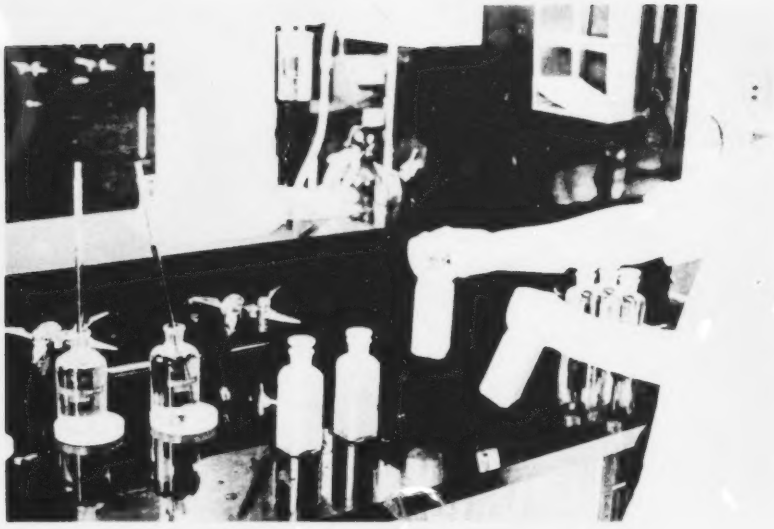


Figure 12-8 Stopper and shake vigorously.

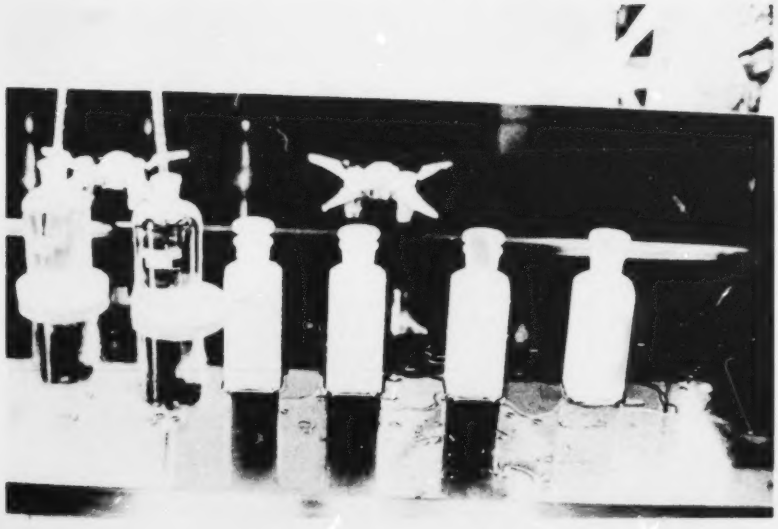


Figure 12-9 Allow mixed samples to settle.

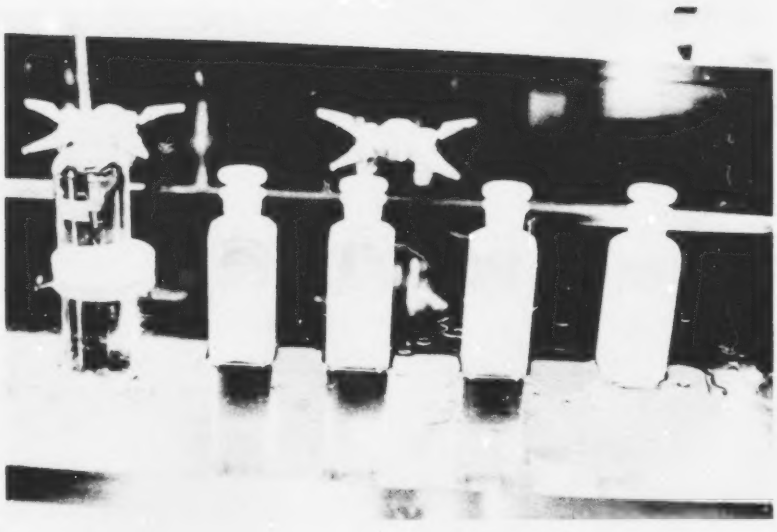


Figure 12-10 Floc allowed to settle for 50% of volume.

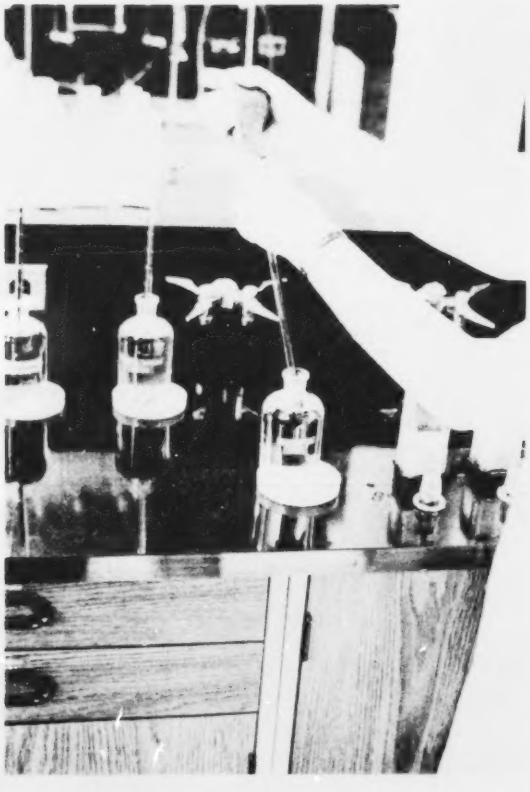


Figure 12-11 Pipetting of Acid.

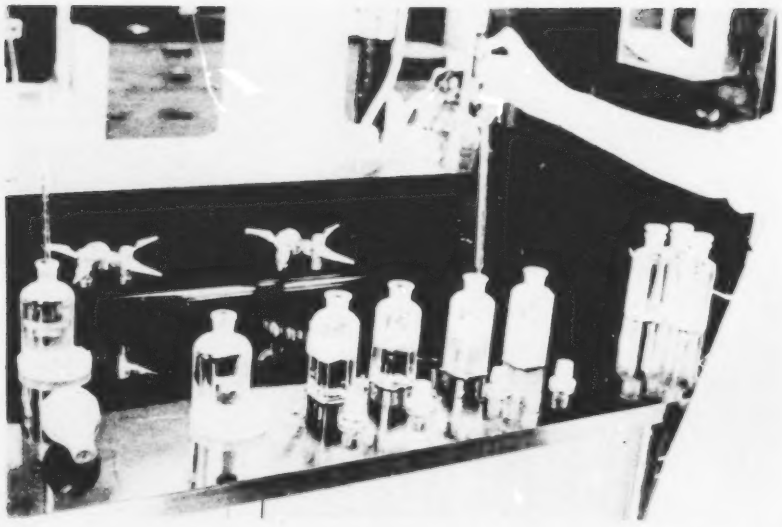


Figure 12-13 Acid addition.

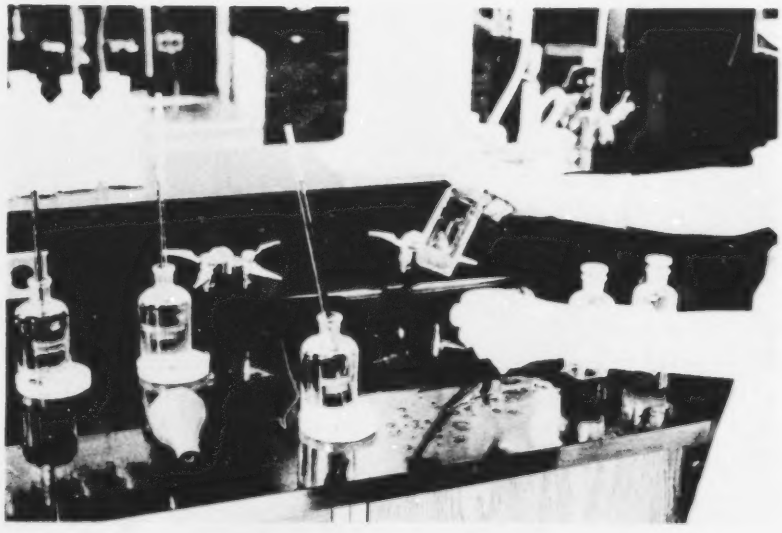


Figure 12-14 Shake to mix and dissolve floc.

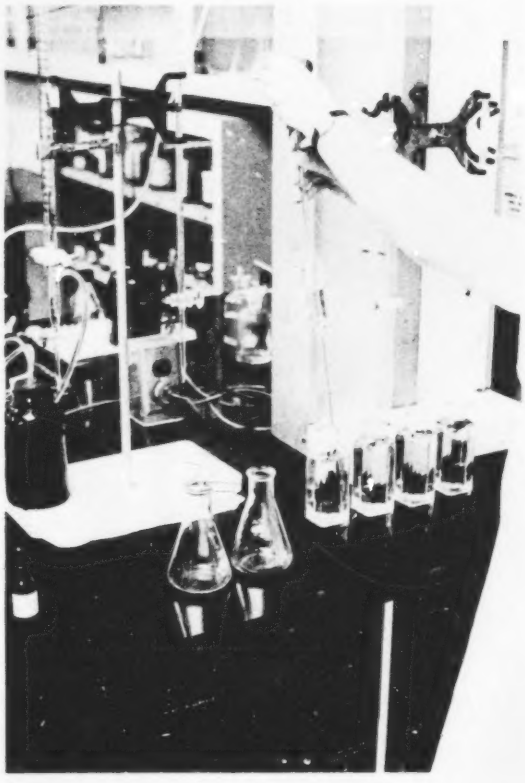


Figure 12-15
Pipetting aliquot for titration.

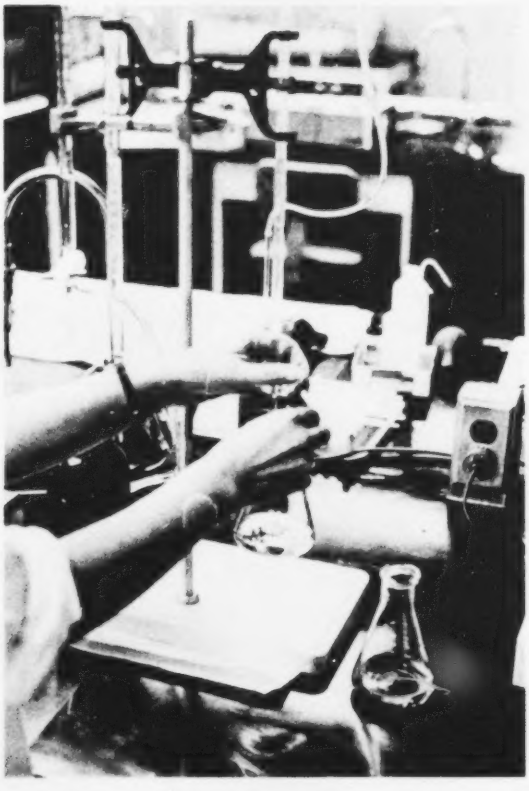


Figure 12-16
Titration of technique.

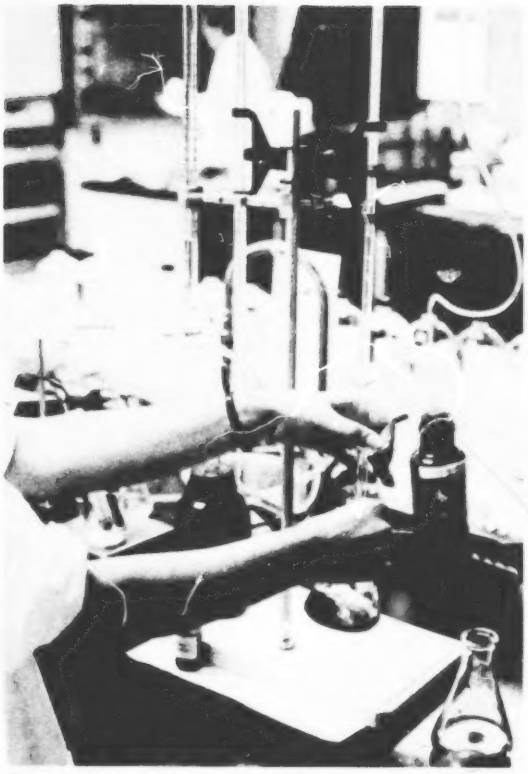


Figure 12-17
Starch addition.

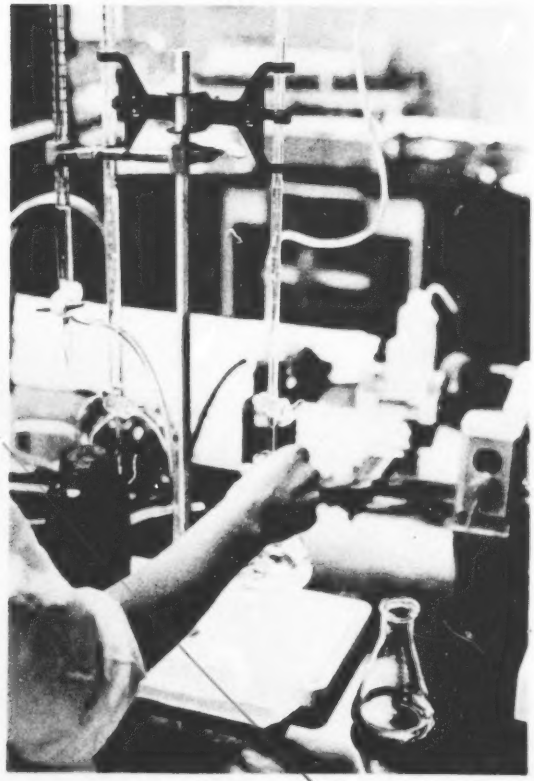


Figure 12-18
End point - End of titration.

SUBJECT:

ACTIVATED SLUDGE
PROCESS CONTROL

TOPIC: 13

OXYGEN UPTAKE TEST

OBJECTIVES:

The trainee will be able to

1. Recall the purpose of the Oxygen Uptake Test.

2. Define Specific Uptake Rate. (SUR)

3. Given the Oxygen Uptake Test results, calculate -

   a) Oxygen Uptake Rate

   b) Specific Uptake Rate (SUR)

OXYGEN UPTAKE TEST

Purpose

Biological waste treatment in the activated sludge process is based on the ability of micro-organisms to utilize dissolved oxygen in breaking down soluble organic substances.

The oxygen uptake test is a means of measuring the respiration rate of the organisms in the activated sludge process. Since it measures the oxygen used in the process, it is a useful tool in the evaluation of process performance, aeration equipment and biodegradability of the waste. So that comparisons can be made between various plants, it is usually expressed as the SUR (specific uptake rate); i.e. the amount of oxygen in mg utilized by one gram of the volatile suspended solids in the activated sludge, in one hour.

Equipment Required

1. Dissolved oxygen meter
2. 1 litre Erlenmeyer flask with stopper suitably bored to admit oxygen probe
3. Magnetic stirrer and magnetic stirring bar
4. 5 litre aspirator bottle or other suitable container.

Procedure for Determining the Uptake Rate

1. Using the dissolved oxygen meter measure and record the DO and temperature of the activated sludge in the aeration tank at the effluent end.
2. Take approximately 2 litre of the mixed liquor and place it in the aspirator bottle.
3. Place the Erlenmeyer flask with the magnet inside it on top of the magnetic stirrer.
4. Shake the activated sludge in the aspirator bottle vigorously for about 15 seconds and fill the Erlenmeyer flask almost to the top.
5. Place the DO probe in the flask making sure the

stopper provides a good seal and switch on the magnetic stirrer.

6. After about 1 to 2 minutes record the DO concentration in the flask every minute for approximately 10-20 minutes. The actual time required depends on the rate of oxygen depletion. Allow sufficient time to get <u>at least</u> 1 mg/l DO difference between start and finish of the test.
7. Plot the oxygen depletion against time on the "Oxygen Utilization Test Sheet" and calculate the uptake rate.

<u>Calculation of Oxygen Uptake Rate (Refer to Oxygen Utilization Test Sheet, P.13-3)</u>

1. The dissolved oxygen values recorded every minute are plotted on the graph of the Test Sheet.
2. A straight line is then drawn so that it passes through the most number of points.
3. The slope can now be calculated: two points in time are selected through which the line passes (e.g. 0 minutes and 10 minutes are used in the example sheet). The DO at these points in time is then observed (e.g. 7.4 mg/l at 0 minutes and 3.4 mg/l at 10 minutes). By subtracting the lower DO value from the higher and dividing the answer by the time interval selected, the slope is obtained.

   Thus $\frac{7.4 - 3.4}{10} = 0.4$ mg O_2/l/min.
4. The Uptake Rate is expressed normally in the units mg O_2/l/hr therefore from the slope:

   Uptake Rate = 0.4 mg O_2/l/min x 60 min/hr
   = 24 mg O_2/l/hr.
5. The Specific Uptake Rate (SUR) can now be calculated:

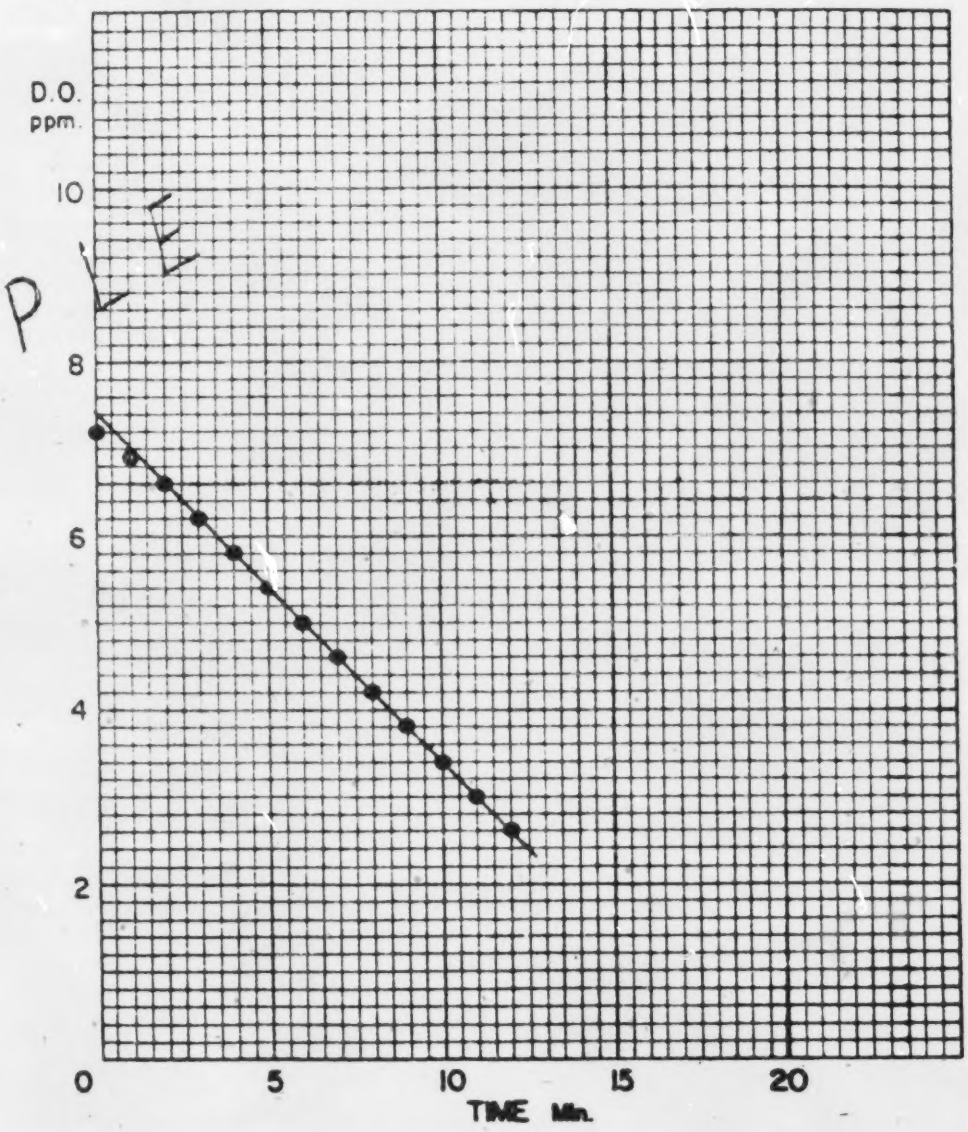
$$SUR = \frac{\text{Uptake Rate} \times 1000}{\text{MLVSS (ppm)}}$$

OXYGEN UTILIZATION TEST NO. _____ DATE: _____

LOCATION :	Bugtown WPCP
TEST CONDITIONS :	Aeration tank No.2. East Side. Initial DO 2.3 ppm
TEMPERATURE :	18°C
SLOPE :	$\frac{7.4 - 3.4}{10}$ = 0.4 mg/l/min
UTILIZATION RATE :	24 mg/l/hr

TIME Start	TIME Min	D.O ppm		
2.00 pm	0	7.2		
	1	6.9		
	2	6.6		
	3	6.2		
TEMP.°C	4	5.8		
	5	5.4		
	6	5.0		
	7	4.6		
18°C	8	4.2		
	9	3.8		
	10	3.4		
	11	3.0		
	12	2.6		

EXAMPLE

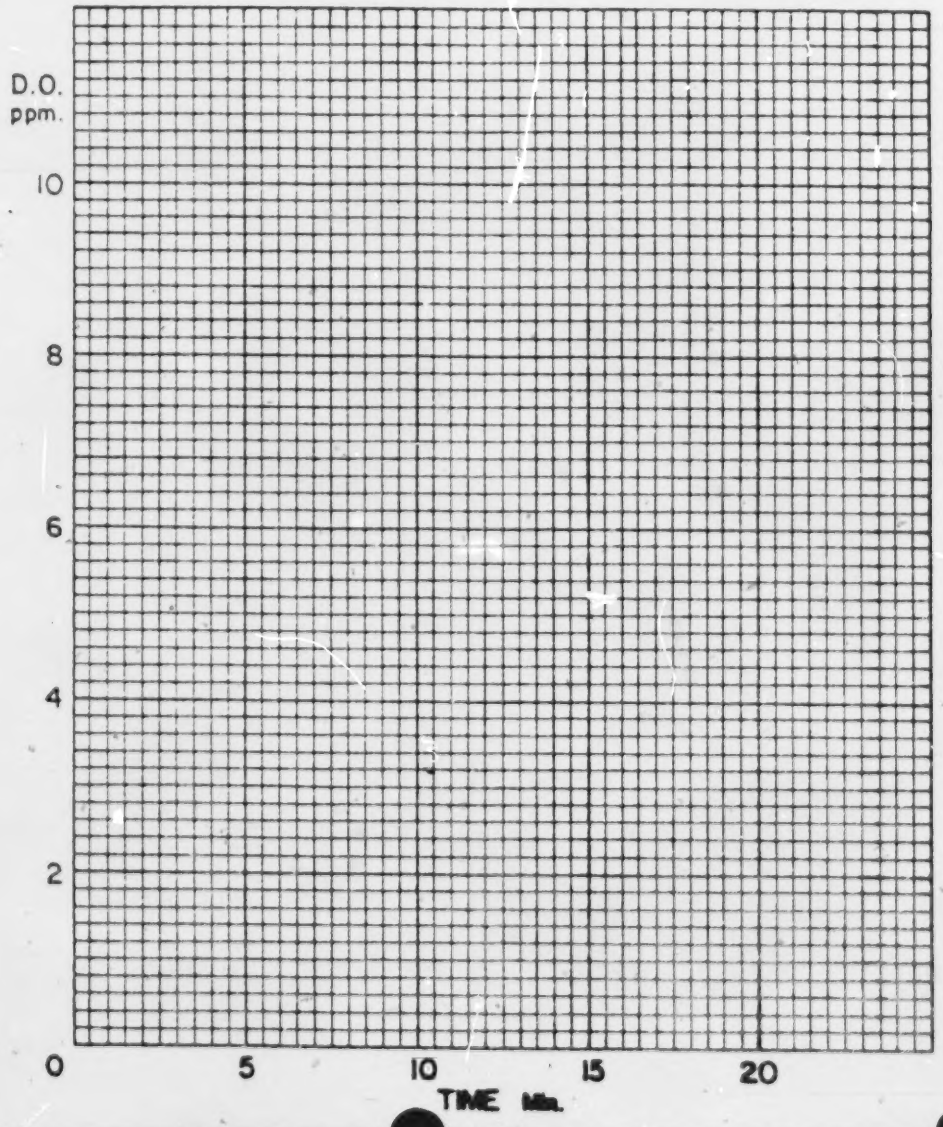


13-3

OXYGEN UTILIZATION TEST NO. _____ DATE: _____

LOCATION :
TEST CONDITIONS :
TEMPERATURE :
SLOPE :
UTILIZATION RATE :

TIME Start.	TIME Min	D.O. ppm.		
TEMP. °C				



e.g., say volatile suspended solids = 2400 ppm*
and uptake rate = 24 mg/l/hr

$$SUR = \frac{24 \times 1000}{2400} = 10 \text{ mg } O_2/l/hr/g \text{ MLVSS}$$

*This figure would have been obtained from the routine daily solids analysis.

Application in Process Control

Once the operator has established the SUR for his own process he can then recognize some upsets which may occur even before the effluent quality is affected.

Example A:

Suppose that at 10:00 a.m. the DO in the aeration tanks is normally 3.4 mg/l and the SUR is 6 mg O_2/l/hr/g. However, although everything looks normal a second measurement around 3:00 p.m. shows that the DO is only 0.5 mg/l and the SUR 14 mg O_s/l/hr/g.

These conditions are indicative that an *abnormally high* organic load has entered the plant. In order to avoid poor effluent quality, the DO in the aeration tanks should be increased by employing additional blowers, or by increasing the immersion or speed of the mechanical aerators. At the same time, samples would be taken and efforts should be made to trace the origin of the abnormal organic load, to determine the type of wastes. The local office of the Ministry of the Environment should be consulted.

Substances which inhibit biological processes, usually metal wastes from industry, will cause a substantial decrease in the uptake rate and an increase in the initial DO.

Example B:

Let us consider the same plant at 10:00 a.m.; the DO is 8 mg/l and the SUR is 2 mg O_2/l/hr/g. Everything else is normal.

These conditions are indicative of a substantial *decrease* in biological activity, caused by a toxic substance which has entered the plant since the last uptake test was done, or a substantial decrease in the organic content of the sewage has occurred. This latter case is unlikely however.

Little can be done to save the effluent quality, and with low biological activity it is going to worsen rapidly. Samples of the aeration tank contents and raw sewage should be collected and all efforts should be made to trace the origin of the toxic substance such as by determining the type of wastes. The nearest office of the Ministry of the Environment should be notified so that further action can be taken.

This technique will give an operator some control and knowledge of his biological process and possibly of accidental spillages to the sewer coming from a particular company or industry. The accumulated SUR data combined with other operating data are invaluable to district staff in assessing plant performance and capability.

Other Applications

Although not of direct interest to the plant operator, the oxygen uptake test can be used further in waste treatment technology.

1. Measurement of the oxygen uptake rates in conjunction with power measurements on the aeration device efficiencies. This is known as the "Steady State Method" of aerator evaluation and is used in design and process evaluations.
2. In conjunction with laboratory or pilot scale aeration tanks, respiration rates can be used to assess the biodegradability of industrial wastes or the effect of industrial wastes on an existing system.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 14

BOD TEST

OBJECTIVES:

The trainee will be able to

1. Recall the purpose of the BOD test.
2. Describe in general terms how BOD test is performed.

BOD TEST

GENERAL

The biochemical oxygen demand (BOD) of a wastewater effluent or polluted water is determined using the dilution technique described in the 13th edition of *Standard Methods*. The test is performed using a five day incubation period and is therefore most often referred to as the 5-day BOD or BOD_5 simply BOD.

The demand for oxygen is determined by measuring the dissolved oxygen content of a sample diluted with specially prepared water containing only mineral nutrients at a certain time and measuring the dissolved oxygen content of that same sample dilution again after five days. The difference in dissolved oxygen concentration, called depletion, is assumed to be due to the biochemical action of microorganisms on the organic nutrients available in the sample. A simple calculation involving the two measured dissolved oxygen concentrations and the known per cent dilution will yield the BOD value.

PURPOSE

The BOD values obtained are derived from analyses performed at empirical (artificial) laboratory test conditions which, over the years, have become standardized. The data obtained are most useful as indications of plant performance in its entirety or individual stages. Another application might be called wastewater characterization where unusually high or low BOD values could reflect on the presence of high "strength" wastes or wastes not biologically treatable. Regular checking of the final effluent BOD is required to ensure that effluent quality meets the objectives set by the Ministry of the Environment.

APPARATUS

Apparatus required to do the BOD determinations includes the following:

1. BOD bottles (300 ml) with ground glass stoppers (number of bottles initially 5 times the number of weekly samples)

2. Incubator, thermostatically controlled to 20°C ± 1°C

3. Graduated cylinders (2 of each):
   1,000 ml capacity, with polyethylene stopper
   250 ml capacity

4. Pipets: (widemouth type) (2 of each)
   10 ml graduated type
   25, 50, 100 ml volumetric type

5. Reagent bottles
   4 of 1-litre capacity with ground glass stoppers
   1 250-ml with ground glass stopper
   1 250-ml, polyethylene with screw cap

6. Miscellaneous:
   Selection of beakers for sample neutralization and dechlorination

   One litre volumetric flask for reagent preparation

   Optional:
   Magnetic stirring apparatus
   pH meter
   Balance for reagent preparation

7. Apparatus and reagents for DISSOLVED OXYGEN DETERMINATION (See Topic 8)

8. Vessel for preparation of BOD water. Size will depend on number of BOD tests normally performed. Vessel can be either glass or plastic.

REFERENCES

1. *Standard Methods for the Examination of Water and Wastewater* - Thirteenth Edition

2. *Simplified Laboratory Procedures for Wastewater Examination* WPCF Publication #18

3. *Methods for Chemical Analysis of Water and Wastes 1971 EPA* - WQO

REAGENT PREPARATION

Distilled Water

Water used for solutions and for preparation of dilution water must be of the highest quality, distilled from a block tin or all-glass still; it must be free of chlorine, chloramines, caustic alkalinity, organic material or acids.

Phosphate Buffer Solution

Dissolve 8.5 g potassium dihydrogen phosphate, (KH_2PO_4), 21.75 g dipotassium hydrogen phosphate (K_2HPO_4), 33.4 g disodium hydrogen phosphate heptahydrate ($Na_2HPO_4.7H_2O$), and 1.7 g ammonium chloride (NH_4Cl) in about 500 ml distilled water and dilute to 1 litre. Discard the reagent (or any of the following reagents) if there is any sign of biological growth in the stock bottle. (Available from Fisher Scientific, Reagent So-P-34).

Magnesium Sulphate Solution

Dissolve 22.5 g magnesium sulphate ($MgSO_4.7H_2O$) in distilled water and dilute to 1 litre. (Available from Fisher Scientific, Reagent So-M-109).

Calcium Chloride Solution

Dissolve 27.5 g anhydrous calcium chloride ($CaCl_2$) in distilled water and dilute to 1 litre. (Available from Fisher Scientific, Reagent So-C-10).

Ferric Chloride Solution

Dissolve 0.25 g ferric chloride ($FeCl_3.6H_2O$) in distilled water and dilute to 1 litre (Available from Fisher Scientific, Reagent So-F-97).

Acid Solutions

1 N sulphuric acid: *carefully* add 28 ml of concentrated sulphuric acid to about 500 ml distilled water with constant stirring. Dilute to 1 litre with distilled water.

Alkali Solutions

1 N sodium hydroxide: carefully add 67 ml of 50% W/W sodium hydroxide solution to about 500 ml distilled water with constant stirring. Dilute to 1 litre with distilled water and store in a polyethylene bottle.

NOTE: DO NOT USE GLASS STOPPERS IN GLASS BOTTLES WITH THIS SOLUTION.

Sodium Sulphite Solution (approximately 0.025N)

Dissolve 1.575 g anhydrous sodium sulphite (Na_2SO_3) in 1 litre distilled water. This solution is not stable and should be prepared as required.

Potassium Iodide

Crystalline KI, reagent grade.

TEST PREPARATIONS

Preparation of the BOD Dilution Water

1. Store the necessary quantity of distilled water in an incubator at 20°C overnight. For example, store 2 litres of distilled water per sample for 2 dilutions.
2. The following morning, saturate the distilled water with atmospheric oxygen by doing the following: fill 1-gallon jugs approximately 3/4 full. Shake for 3 to 4 minutes, or use a magnetic stirrer apparatus if available.
3. Add 1 ml of phosphate buffer, 1 ml of magnesium sulphate, 1 ml of calcium chloride and 1 ml of ferric chloride solution for each litre of water that is prepared. Shake vigorously for approximately 2 minutes to assure complete mixing, or use a magnetic stirrer apparatus if available.

Sample Pretreatment - pH Adjustment

1. If sample pH is ABOVE 8.5, neutralize sample by adding 1 N sulphuric acid in the following manner: Place 100 ml of sample in a 250 ml beaker. Add 1 N sulphuric acid drop by drop and mix well by using a clean glass stirring rod, or use a magnetic stirrer apparatus if available.
   RECHECK THE pH AFTER EACH DROP OF CHEMICAL ADDITION UNTIL pH IS BETWEEN 6.8 AND 7.2.

2. If sample pH is BELOW 6.0, repeat step 1 using 1 N sodium hydroxide instead of 1N sulphuric acid.
   RECHECK pH AFTER EACH DROP OF CHEMICAL ADDITION UNTIL pH IS BETWEEN 6.8 AND 7.2.

Sample Pretreatment - Removal of Residual Chlorine

If a sample contains residual chlorine, as in a chlorinated final effluent, dechlorinate as follows:

1. Measure out 100 ml of sample into a 250 ml Erlenmeyer flask.

2. Add 0.5 ml of concentrated sulphuric acid. Mix well by swirling.

3. Add 1 g potassium iodide. Mix well by swirling until all potassium iodide is dissolved.

4. a) If a YELLOW COLOUR develops, chlorine residual is present. Proceed to step 5.

   b) If NO YELLOW COLOUR develops, chlorine residual is not present in sample. Proceed with "Sample Dilution Technique and Incubation", page 14-7.

5. If yellow colour is present, use freshly prepared 0.025N (approx.) sodium sulphite and titrate the 100 ml sample in the Erlenmeyer flask, following the same procedure used in the WINKLER DISSOLVED OXYGEN TITRATION (see Dissolved Oxygen Determination). Use starch solution as an end-point indicator. Record the titration volume.

6. Having determined the titration volume of sodium sulphite solution required to dechlorinate 100 ml of sample, the following formula will determine the required amount of sodium sulphite solution for any volume of sample:

$$V = \frac{a \times b}{100}$$

where V = volume of titrant required to dechlorinate new sample aliquot

a = volume of sample to be dechlorinated

b = volume of sodium sulphite solution found necessary to dechlorinate 100 ml of sample

7. Allow 5 minutes before proceeding with "Sample Seeding".

Sample Seeding

Samples that have required pH adjustment or dechlorination may not contain sufficient "healthy" bacteria to start a BOD reaction. For this reason, a small volume of stale domestic raw sewage is introduced into the dilution selected for the sample to provide an inoculum of bacteria. With municipal wastewaters, seeding is normally not required.

To simplify later calculations a seed concentration of 1/10 the sample dilution is used; e.g. for a 5% sample dilution, a 0.5% seed concentration would be used.

1. Add a known percentage of seeding material (domestic raw sewage, settled overnight) to the sample dilution. (See dilution technique, page 14-7, to determine the number of sample dilution required.)

2. Apply the mathematical correction for the presence of the seed material in the sample dilution to the BOD calculation (see page 14-11).

Sample Dilution Technique and Incubation

The appearance of the sample is a good indicator for estimating the best dilutions that will yield satisfactory BOD results. Those samples higher in solids will usually have a higher BOD; for example, a raw sewage composite sample normally has a higher BOD than the corresponding primary effluent composite sample. Final effluents that are low in suspended solids (15 mg/l or less) are reasonably free of turbidity and will usually have low BOD values of 15 mg/l or less . In domestic sewage, the major contribution of the BOD is caused by particulate suspended organic matter and a lesser portion is contributed by dissolved organic matter. The suspended solids concentration is therefore often a good guideline for estimating the proper dilutions in the BOD test.

1. For each sample, prepare 2 bottles for dilution; if the sample is unknown, prepare at least 3 bottles for dilution.

   a) If using the Winkler Method for the dissolved oxygen determination, prepare 2 bottles for each sample dilution, one for immediate dissolved oxygen determination, the other one for dissolved oxygen determination after 5 days of incubation.

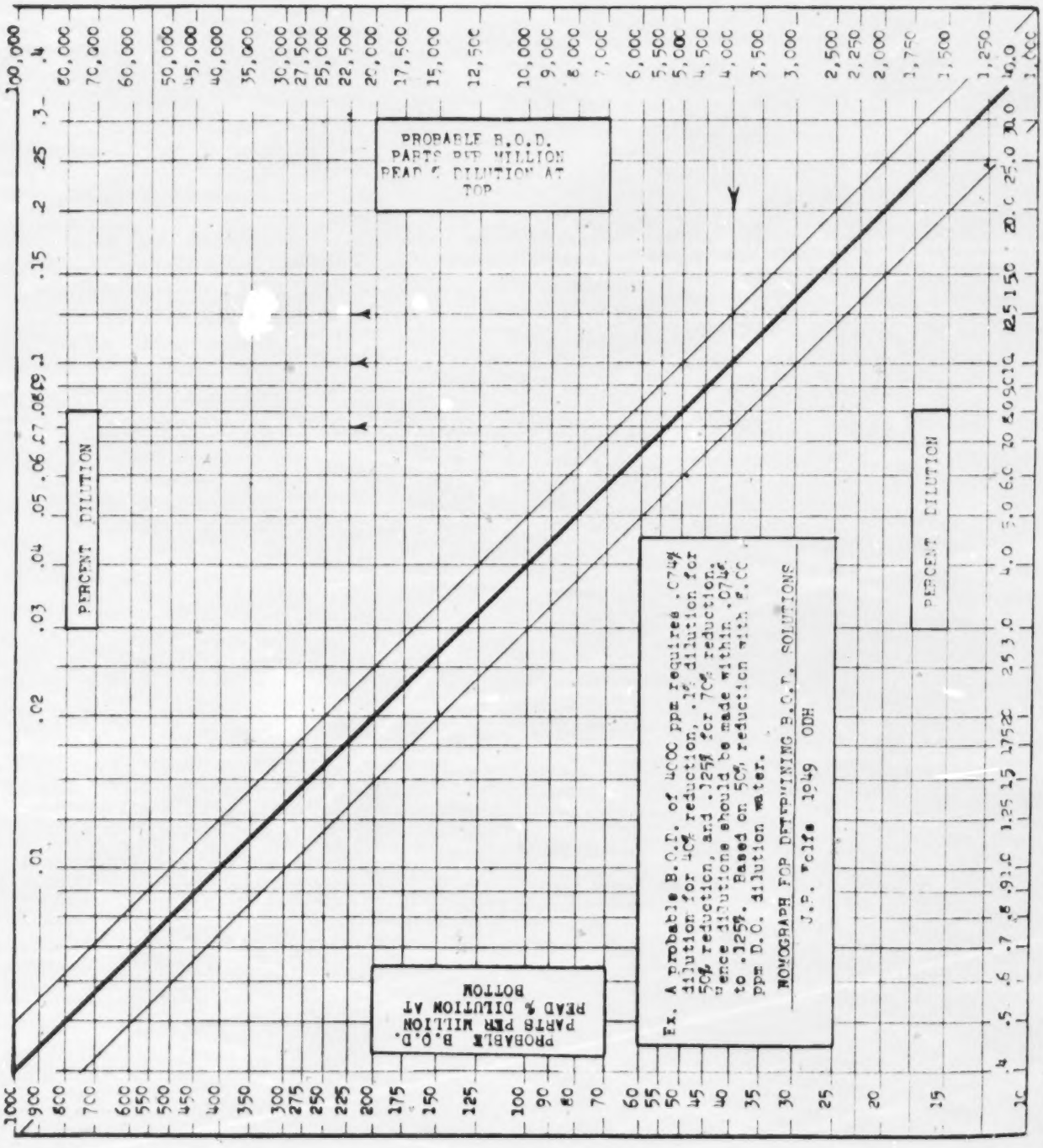
   b) If a dissolved oxygen meter is used for dissolved oxygen determination, prepare only one bottle for each dilution.

2. The following table of BOD ranges can be useful in arriving at proper BOD dilutions along with the nomograph (Figure 14-1) on page 14-8.

TABLE 14-1 - BOD RANGE vs TYPE OF WASTE

Type of Waste	BOD RANGE (mg/l)		
	weak	medium	strong
raw sewage	50-100	150-250	250-400 or more
primary effluent	35-70	100-200	200-300
final effluent	good	poor	very poor
	less than 5-15	15-50	greater than 50

NOMOGRAPH FOR DETERMINING PROPER DILUTION IN THE B.O.D. TEST


PROBABLE B.O.D.
PARTS PER MILLION
READ % DILUTION AT
TOP
PERCENT DILUTION
PERCENT DILUTION
PROBABLE B.O.D.
PARTS PER MILLION
READ % DILUTION AT
BOTTOM
Ex. A probable B.O.D. of 4000 ppm requires .07% dilution for 40% reduction, .1% dilution for 50% reduction, and .125% for 70% reduction. Hence dilutions should be made within .07% to .125%. Based on 5% reduction with 8.CC ppm D.O. dilution water.
NOMOGRAPH FOR DETERMINING B.O.D. SOLUTIONS
J.R. Wolfe 1949 ODH

In making the Biochemical Oxygen Demand Test it is desirable to make up the dilution of the sample so that not more than 70% nor less than 40% of the dissolved oxygen in the diluted sample will be used up during the incubation period of 5 days. As an aid in choosing the proper dilutions for the 5-day B.O.D. Test the above nomograph has been developed by John R. Wolfe, Ass't. Engr., Ohio State Dept. of Health

Example Using Nomograph

Suppose the raw sewage is of medium "strength" as determined by visual inspection and possibly from a suspended solids analysis. The BOD is estimated to be around 200. According to the nomograph, a BOD of 200 requires a dilution of 2%. Follow the line at 200 across to the thick black line, then follow the intersected line to arrive at 2.0 per cent dilution. The line at 2% crosses the two thin black lines of the graph at BOD values of 150 and 250. This means a 2% dilution can be used reliably for a BOD range of 150 to 250. To protect against errors in BOD estimation, one or two more dilutions should be set up to take care of unexpected high or low values. This is also a good way to double check the result obtained for the first dilution. Dilutions of 1.25% and 3% would cover the BOD range from 100 to 400 and setting up these additional dilutions is recommended in this case.

PROCEDURE FOR TEST

1. Neutralize and/or dechlorinate an aliquot of the sample requiring BOD determination.

2. For each pretreated sample select at least two dilutions using the Table 14-1 and accompanying nomograph.

3. For each sample dilution chosen, label two clean BOD bottles for Winkler Dissolved Oxygen Method or one BOD bottle for dissolved oxygen meter method . The label should identify the sample, the per cent dilution chosen and the date on which the sample is to be removed from the incubator.

   To simplify the preparation of the dilutions and to minimize errors *always prepare one litre of sample dilution, discarding the unused portion.*

4. Using a volumetric pipet for volumes up to 100 ml, or a graduated cylinder for volumes larger than 100 ml, add the selected sample aliquot to a 1,000 ml graduated cylinder that is fitted with a polyethylene stopper. Add BOD dilution water to the

1,000 ml mark. Stopper the cylinder and turn it upside down slowly four or five times to mix the contents. Remove the stopper and completely fill two properly labelled BOD bottles with the sample dilution by pouring it slowly down the inside neck of each bottle held slightly tipped, just as one would pour beer into a glass to prevent foaming. Discard the unused portion of the sample dilution.

5. Prepare a second and third (if required) dilution of the same sample as in above.

6. Wait 15 minutes after filling the BOD bottles with sample before stoppering them. WHY? To allow any bubbles to rise to the top of the bottle. Bubbles seen settling on the inside shoulder of the bottle should be removed by grasping the bottle by its neck and gently tapping the outside shoulder of the bottle with the glass stopper.

7. Stopper the bottles with the glass stoppers. Avoid entrapment of air by inclining the bottle slightly and inserting the stopper with a quick motion.

8. a) Using the Winkler Method, determine the dissolved oxygen concentration on one bottle of each sample dilution that has been prepared as soon as the bottles have been stoppered. Place the other dilution bottles in an air incubator for five days at $20^{\circ}C \pm 1^{\circ}C$ ($68^{\circ}F \pm 2^{\circ}F$).

   b) *If dissolved oxygen meter with BOD bottle probe is used*, only one bottle of each sample dilution is prepared. In this case, the dissolved oxygen content is determined as soon as the sample dilution is ready to be stoppered. After the dissolved oxygen content has been determined and recorded, the bottle is stoppered, taking care to prevent entrapment of air bubbles. The bottle is then incubated for five days at $20^{\circ}C \pm 1^{\circ}C$ and the oxygen content redetermined.

9. Dilutions showing a residual dissolved oxygen concentration of at least 1 mg/l and a depletion of at least 2 mg/l should be considered the most reliable. The precision of the test is about ± 20%. This means that for a sample having a BOD of 200, the test may show results as high as 240 or as low as 160. Keep this in mind when relatively poor correlation is found in a particular test series. Averaging of the most reliable results obtained in the test on a particular sample may be used.

CALCULATIONS

$$\text{mg/l BOD} = \frac{(D_1 - D_2) \times 100}{d}$$

where D_1 = dissolved oxygen content (mg/l) of sample dilution 15 minutes after preparation.

D_2 = dissolved oxygen content (mg/l) of sample dilution after 5-day incubation.

d = per cent dilution of the sample used.

Where the sample has been seeded, the following formula is used:

$$\text{mg/l BOD} = \left[(D_1 - D_2) - (S_1 - S_2)f\right] \times 100$$

where D_1 = dissolved oxygen content (mg/l) of sample dilution 15 minutes after preparation.

D_2 = dissolved oxygen content (mg/l) of sample dilution after 5-day incubation.

S_1 = dissolved oxygen content of seed control dilution 15 minutes after preparation.

S_2 = dissolved oxygen content of seed control after 5- day incubation.

f = ratio of % seed used in sample (D_1) to % seed used in control (S_1).

EXAMPLE 1

A raw sewage sample BOD determination was performed using dilutions of 1%, 2% and 5%. The following results were found:

d	D_1	D_2	$D_1 - D_2$	
1%	8.4	6.0	2.4	$\frac{2.4 \times 100}{1} = 240$
2%	8.3	4.2	4.1	$\frac{4.1 \times 100}{2} = 205$
5%	8.2	0.4	7.8	$\frac{7.8 \times 100}{5} = 156$

The 1% and 2% dilution show depletions ($D_1 - D_2$) of more than 2 mg/l and residual dissolved oxygen levels (D_2) of more than 1 mg/l. The 5% dilution shows a residual dissolved oxygen level of less than 1 mg/l, making the result unreliable and it should not be used. The other two results which are calculated to be 240 and 205 are reliable. The average of the two results to 222.5. This number should be rounded off to 220 when reporting the result.

EXAMPLE 2

A sample of chlorinated final effluent was dechlorinated using the prescribed method and stale raw sewage was used to seed the sample.

The sample was seeded with 0.3% of stale sewage and the seed control was incubated at 3% concentration. The following results were found on a 25% seeded sample dilution and the seed control.

D_1 = 8.3 ppm D.O.

D_2 = 4.5 ppm D.O.

S_1 = 8.0 ppm D.O.

S_2 = 3.1 ppm D.O.

Enter these results in the BOD equation:

$$\text{mg/l BOD} = \frac{(8.3 - 4.5) - (8.0 - 3.1)\ \frac{0.3}{3}}{0.01 \times 25}$$

$$= \frac{(3.8) - (4.9)\ 0.1}{0.25}$$

$$= \frac{3.8 - 0.49}{0.25}$$

$$= \frac{3.31}{0.25}$$

$$= 13.2$$

The result is reported as 13 mg/l.

SUBJECT:

ACTIVATED SLUDGE PROCESS CONTROL

TOPIC: 15

PHOSPHORUS DETERMINATION

OBJECTIVES:

The trainee will be able to

1. Recall the major steps for phosphorus determination.

2. Given the test results, calculate -

   a) soluble orthophosphorus

   b) total orthophosphorus

PHOSPHORUS DETERMINATION

GENERAL

This is the method described in the 13th edition of *Standard Methods* with some small changes in the digestion step and selection of filter paper for the "soluble" phosphorus determination. The term "phosphorus" will be used throughout the description of the method, because of the variety of forms in which phosphorus is chemically bound. All forms are reported as equivalent P.

The 13th edition of *Standard Methods* includes a table, Classification of Phosphate Fractions, where twelve different phosphorus fractions are listed. The two most frequently used fractions are the Total Dissolved and Suspended Phosphate and the Filterable (dissolved) Ortho-Phosphate. The method described here will analyze these two fractions, referring to the former as "Total phosphorus" and to the latter as "Soluble orthophosphorus".

There are three major steps as shown in the flow chart (Figure 15-1) on page 15-2.

1. *Sample Preparation* separates the samples into two groups, depending on which phosphorus fraction is to be determined.
2. *Colorimetry* chemically combines the phosphorus into a highly coloured *molybdenum blue* complex.
3. *Spectrophotometry* determines the intensity or strength of the blue colour by measuring the absorbance of the complex at a specified wavelength. A simple calculation will convert this measurement into the phosphorus concentration of the sample.

APPARATUS

1. Spectrophotometer for use at approximately 690 mμ, accommodating cells with a light path of 1 cm or longer.

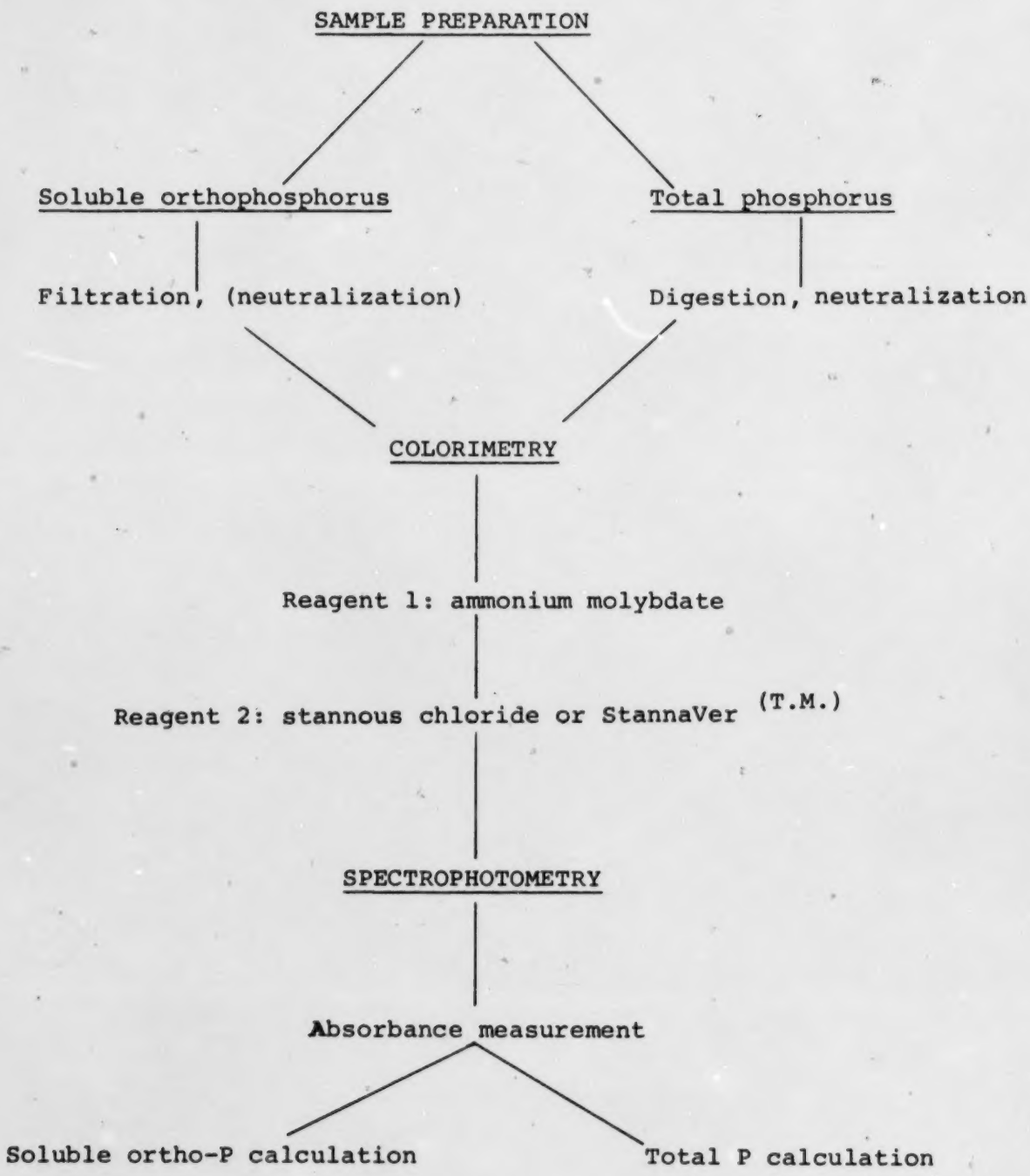


Figure 15-1

PHOSPHORUS DETERMINATION FLOW CHART

2. Nessler tubes graduated at 50 and 100 ml in an appropriate stand or 100 ml graduated cylinders, tall form

3. Glass filter funnels, short stem

4. Glass fibre filter paper, 9.0 cm

5. Hot plate with heat control (about 12 x 12 in. surface)

6. 125 ml Erlenmeyer flasks

7. Pipets:

   - 5, 10, 25, 50 ml transfer pipets
   - 5, 10, ml graduated pipets
   - 5, or 10 ml wide mouth graduated pipets
   - pipet tray or stand

8. Miscellaneous items such as tongs, spatula, wash bottle, Parafilm, glass stirring rods, clippers, dropper bottle

9. Reagent bottles:

   - 3 250 ml clear reagent bottles with ground glass stopper
   - 3 1 litre clear reagent bottles with ground glass stopper
   - 1 1 litre low Actinic reagent bottle with ground glass stopper
   - 1 1 litre polyethylene bottle with screw cap

10. Volumetric glassware:
    - 1 250 ml graduated cylinder
    - 1 1 litre graduated cylinder
    - 2 1 litre volumetric flasks
    - 1 1 litre beaker

11. Scoop to dispense 0.8 g potassium persulphate

REAGENT PREPARATION

1. Phenolphthalein Indicator Solution

   Dissolve 1 g phenolphthalein in 100 ml ethyl alcohol. Add 100 ml distilled water and 0.02N (normal) NaOH with a dropper until a faint pink colour appears. Store the solution in a 250 ml stoppered reagent bottle and dispense the necessary reagent from a glass dropper bottle.

2. 5N Sulphuric Acid

   To approximately 500 ml distilled water, add slowly and with stirring 140 ml concentrated reagent sulphuric acid. Cool, and dilute to one litre reagent bottle.

3. 2N Sodium Hydroxide

   To 800 ml distilled water add slowly with constant stirring 120 ml 50% w/w NaOH solution. Alternatively add 80 g fresh NaOH pellets to 750 ml distilled water and stir to dissolve, cool, dilute to 1 litre. (0.02N NaOH - dilute 1 ml 2N NaOH to 100 ml) Store in a 1 litre polyethylene bottle.

4. Ammonium Molybdate Reagent

   Cautiously add 280 ml concentrated H_2SO_4 to 400 ml distilled water. Cool, and add 25 g ammonium molybdate, $(NH_4)_6MO_7O_{24}.4H_2O$ dissolved in 175 ml distilled water and fill to 1 litre. Store in a brown, low Actinic glass reagent bottle.

5. Stannous Chloride Reagent (I)

   Dissolve 2.5 g of a fresh supply of $SnCl_2.2H_2O$ in 100 ml glycerol. Heat in a water bath and stir with a glass rod to hasten dissolution. Store in a 240 ml reagent bottle. This reagent is stable and requires neither preservative nor special storage.

6. Dilute Stannous Chloride (II)

   Mix 8 ml of stannous chloride reagent (I) with 50 ml glycerol. Use a 10 ml wide mouth graduated pipet. This reagent is stable for at least 6 months.

7. Alternatively to reagents in 5 and 6 use Hach Chemical No. 877 StannaVer 1 lb. lots from:

Hach Chemical Company
713 South Duff
Ames, Iowa, 50010
U.S.A.

8. Anti-bumping Granules

B.D.H. No. 33009 ground glass granules have been found to work well. Ceramic or marble chips may dissolve in the acidic solution and cannot be used.

9. Hydrochloric Acid

Technical or preferably reagent grade for glassware cleaning.

10. Standard Phosphorus Stock Solution

Dissolve 0.4390 g KH_2PO_4 (Potassium dihydrogen orthophosphate) in distilled water and dilute to 1000 ml in a clean volumetric flask. Transfer this stock solution to a clean 1 litre reagent flask, keeping the bottle stored in a dark cupboard. 1.00 ml = 0.100 mg P.

11. Calibration Standard

Using acid cleaned glassware dilute 10.0 ml of the phosphorus stock solution to 1000 ml in a volumetric flask. Prepare this solution fresh for each calibration *preferably reserving the volumetric flask and transfer pipet for this purpose only*, to avoid possible contamination. Use 5, 10, 20, 30, 40, 50, 60, 70 and 80 ml of this solution for calibration purposes. In 100 ml final dilution volume this is equivalent to 0.05, 0.1, 0.2, 0.3, 0.4, 0.5, 0.6, 0.7, and 0.8 mg/l P.

a) Potassium Persulphate

$K_2S_2O_8$, reagent grade.

SAMPLE PREPARATION

Soluble Orthophosphorus

Using glass fiber filter paper fluted into a glass funnel, filter 20 to 50 ml of well shaken sample into a Nessler tube or 100 ml graduated cylinder. In some cases where the soluble orthophosphorus level is exceptionally low up to 90 ml of sample may have to be filtered. Where larger volumes of samples are used it may be necessary to adjust the pH to a value of 7 to overcome the buffering capacity due to the sample's alkalinity. Determine the phosphorus concentration by carrying out the colorimetric procedure described on page 15-7.

Persulfate Digestion for Total Phosphorus

Proper aliquots for the digestion can only be chosen from the appearance of the sample or some previous knowledge that may assist in estimating the approximate phosphorus level. For raw sewage 2, 5 or 10 ml usually suffice; for primary effluents 5 or 10 ml are adequate except where phosphorus removal is performed in the primary clarifier. Depending on treatment efficiency low levels of phosphorus may be encountered, calling for larger sample aliquots. Final effluents vary in total phosphorus concentration depending on treatment method and efficiency of phosphorus removal. 10, 25, or 50 ml will usually suffice in the digestion step.

1. Add 2 ml of 5N sulphuric acid to an aliquot of well mixed sample in a 125 ml Erlenmeyer flask.
2. Using a scoop (calibrated to deliver 0.8 g $\pm$ 0.1 g potassium persulphate) add 0.8 g $K_2S_2O_8$ to the flask. Swirl to mix.
3. Add about half a dozen anti-bumping granules and dilute the sample aliquot in the flask to a total volume of about 35 ml.
4. Boil gently on a preheated hotplate for 30 minutes adding distilled water when necessary to prevent evaporation to dryness.

5. Cool the digested sample to room temperature and filter the digest through glass fiber filter paper if a precipitate has formed during the digestion.

6. Add one drop of phenolphthalein indicator solution to the Erlenmeyer flask and neutralize the digested sample by adding 2N sodium hydroxide solution drop by drop until the pink colour form of the indicator persists. See notes 4 and 5 page 15-10.

7. Transfer the contents of the Erlenmeyer flask to a 100 ml Nessler tube or graduated cylinder.

8. Rinse the flask several times with distilled water and add the rinsings to the tube or cylinder.

9. Add distilled water to the 100 ml mark, cover the tube or cylinder with a piece of Parafilm and invert several times to mix the contents.

10. Determine the phosphorus concentration by means of the colorimetric method described below.

A separate calibration curve must be determined for the total phosphorus test by carrying standards through the persulphate digestion step.

COLORIMETRIC PROCEDURE - STANNOUS CHLORIDE METHOD

1. Measure an appropriate aliquot of sample into a 100 ml Nessler tube or 100 ml graduated cylinder. For soluble orthophosphorus use the filtrate from the sample preparation page 15-6. For total phosphorus use the persulphate digest from the sample preparation page 15-6. For the ortho-phosphorus standardization for total phosphorus transfer the digested calibration standards to 100 ml Nessler tubes or 100 ml graduated cylinders washing the digestion flasks with several portions of distilled water and adding these rinsings to the corresponding Nessler tubes or cylinders.

2. Dilute the sample aliquot or standards to about 90 ml. For best results the temperature of the solution should now be between 20 and 25°C.

3. Add 2 ml of the ammonium molybdate reagent. Invert the tube or cylinder to mix in the reagent, using Parafilm as a cover.

4. After two minutes, add 10 drops of dilute stannous chloride (II) reagent using a wide mouth pipet or alternatively use 0.5 g StannaVer powder (contents of four powder pillows).

5. Dilute with distilled water to 100 ml, cover with Parafilm, invert and shake thoroughly to mix in the reagent. A blue colour ("molybdenum blue") will indicate the presence of phosphorus.

6. After 10 minutes, but before 12 minutes (after the addition of the stannous chloride or StannaVer reagent), measure the absorbance of the coloured solution at 690 mμ. Use distilled water as a blank to set the spectrophotometer to zero absorbance.

7. Wash all glassware used as soon as possible. Rinse first with hydrochloric acid and then with distilled water.

8. Plot the results obtained on the standard solutions on linear graph paper and refer to this graph when calculating results. See example on page 15-9.

Calculation

$$\text{soluble orthophosphorus mg/l} = \frac{\text{mg/l P (read from graph) x 100}}{\text{ml filtered sample used}}$$

$$\text{total phosphorus mg/l} = \frac{\text{mg/l P (read from graph) x 10,000}}{\text{ml sample digested x ml of digest used}}$$

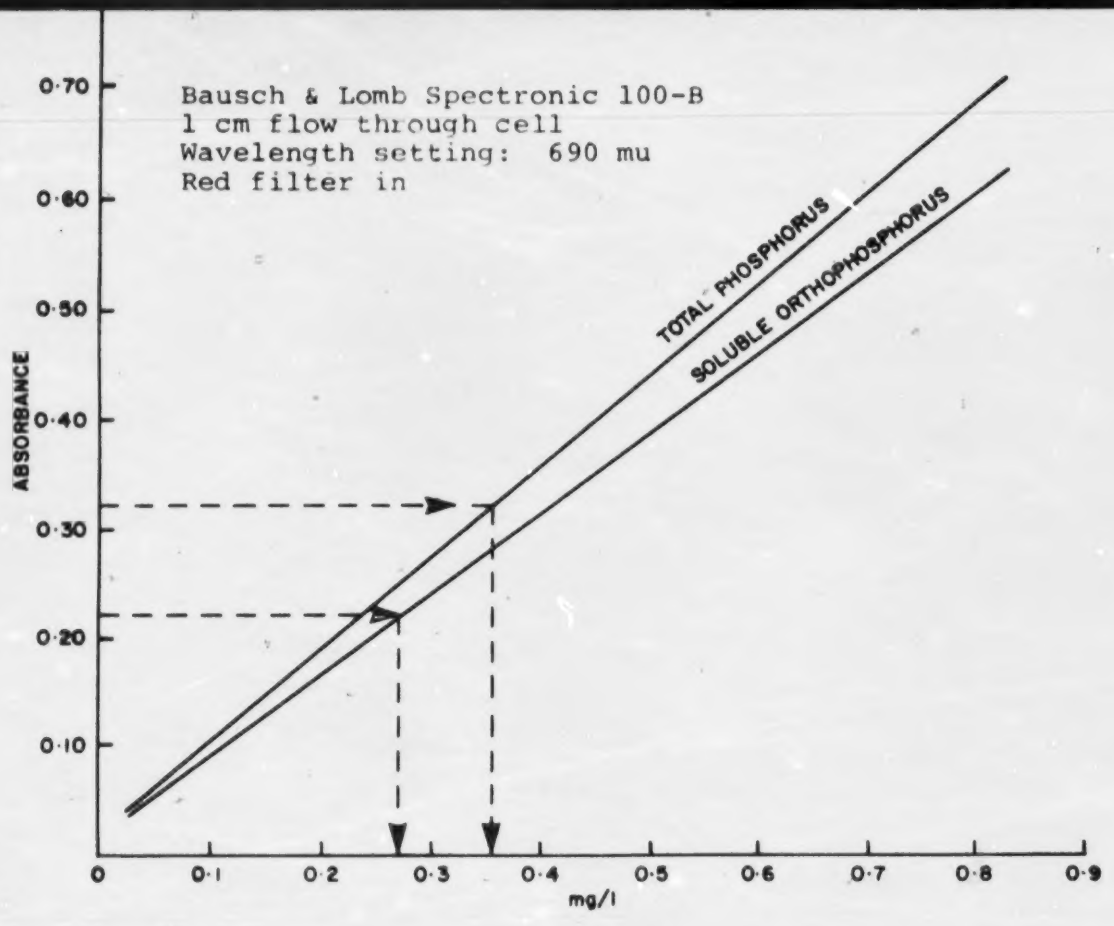


Figure 15-2 TYPICAL PHOSPHORUS DETERMINATION CALIBRATION GRAPH
STANNOUS CHLORIDE METHOD

Sample Calculations:

1. Soluble orthophosphorus: 25 ml sample used
absorbance found was 0.22

From the graph 0.22 absorbance is equivalent to 0.27 mg/l soluble orthophosphorus.

$$\text{mg/l sol. o-p} = \frac{0.27 \times 100}{25} = 1.08 \text{ mg/l sol. o-p}$$

2. Total phosphorus: 10 ml sample digested
50 ml of the diluted digest used
absorbance found was 0.32

From the graph 0.32 absorbance is equivalent to 0.355 mg/l total phosphorus.

$$\text{mg/l total P} = \frac{0.355 \times 10{,}000}{50 \times 10} = 7.1 \text{ mg/l total P}$$

NOTES

1. All steps of the analysis must be performed in the order indicated.
2. For spectrophotometers or colorimeters normally used the most accurate range lies between 0.15 and 0.50 absorbance units. If a measurement falls outside these limits the test should be repeated using either more or less sample aliquot. The situation cannot be remedied by further sample addition or dilution of the coloured complex.
3. If the final colour is yellow, green or brown, the phosphorus concentration may be extremely high (repeat test at lower sample concentration) or serious interference may be present.
4. If in the neutralization step of persulphate digestion a reddish brown precipitate is formed, the digestion should be repeated using a smaller aliquot.
5. Over neutralization by addition of too much sodium hydroxide will produce high results in the colorimetric step. Thus neutralization must be performed exactly.

References

(1) Gales, M.E., et al, Method for Quantitative Determination of Total Phosphorus in Water, American Water Works Association Journal, v. 58 (10): p. 1365, 1966.

(2) Harwood, R.A., A Comparison of Some Methods for Total Phosphate Analyses, Water Research, V. 3: p. 426, 1969.

GLOSSARY OF TERMS

The following definitions are intended only as aids in the study of this manual.

absorption -
The taking up of one substance into the body of another.

activated sludge -
Sludge floc produced in raw or settled wastewater by the growth of zoogleal bacteria and other organisms in the presence of dissolved oxygen and accumulated in sufficient concentration by returning floc previously formed.

adsorption -
(1) The adherence of a gas, liquid, or dissolved material on the surface of a solid.

(2) A change in concentration of gas or solute at the interface of a two-phase system. Should not be confused with absorption.

aeration -
(1) The bringing about of intimate contact between air and a liquid by one or more of the following methods:

a. spraying the liquid in the air,
b. bubbling air through the liquid,
c. agitating the liquid to promote surface absorption of air.

(2) The supplying of air to confined spaces under nappes, downstream from gates in conduits, etc. to relieve low pressures and to replenish air entrained and removed from such confined spaces by flowing water.

(3) Relief of the effects of cavitation by admitting air to the section affected.

aerobic -
Requiring, or not destroyed by, the presence of free elemental oxygen.

algae -
Tiny plants, usually living in water and often green.

alkaline -
A condition which will raise the pH in water or wastewater higher than 7.

bacteria -
Single-celled microscopic plants living in soil, water, organic matter, or the bodies of plants and animals.

baffle -
A device to turn aside, check, or regulate flow.

barminutor -
Trade name for a shredding device.

bar screen -
A rack made of parallel bars for removing coarse materials in the wastewater passing between them.

BOD -
Biochemical Oxygen Demand. A measure of the oxygen used in decomposing organic matter.

bulking -
Bulking occurs in activated sludge plants when the sludge becomes too light and will not settle properly.

centrifuge -
A machine that separates solids from wastewater in a spinning motion.

chemical oxygen demand -
A measure of the oxygen-consuming capacity of inorganic and organic matter present in water or wastewater. It is expressed as the amount of oxygen consumed from a chemical oxidant in a specific test. It does not differentiate between stable and unstable organic matter and thus does not necessarily correlate with biochemical oxygen demand. Also known as OC and DOC, oxygen consumed and dichromate oxygen consumed, respectively.

chlorine demand -
The difference between the amount of chlorine added to a water or wastewater and the amount of chlorine residual left after a certain length of time.

clarifier -
A unit of which the primary purpose is to secure clarification. Usually applied to sedimentation tanks or basins.

coagulants -
In water and wastewater, chemicals used to thicken finely divided suspended solids into groups for easy removal.

coagulation -
In water and wastewater treatment, the destabilization and initial aggregation of colloidal and finely divided suspended matter by the addition of a floc-forming chemical or by biological processes.

colloidal -
Finely divided solids which will not settle but may be removed by coagulation or biochemical action or membrane filtration.

comminutor -
A shredding device used in pretreatment.

cross flight -
Wooden scraper for moving sludge and scum in a rectangular clarifier.

decomposition -
Generally aerobic processes that convert unstable materials into more stable forms by chemical or biological action. Waste treatment encourages decay in a controlled situation in order that the material may be disposed of in a stable form. When organic matter decays under anaerobic conditions (putrefaction), undesirable odours are produced. In aerobic processes, the odours are much less objectionable than those produced by anaerobic decomposition.

detention time -
The length of time that wastewater is held in a unit for treatment.

detritor -
Equipment used in pretreatment to remove heavy minerals such as grit, and other coarse debris carried in water and wastewater.

diffuser -
A device for distributing tiny air bubbles throughout a liquid, such as wastewater.

digestion -
The biological decomposition of organic matter to a more stable form.

dissolved oxygen -
Atmospheric oxygen dissolved in water or wastewater, usually abbreviated DO.

effluent -
In wastewater treatment, wastewater or other liquid, partially or completely treated or in its natural state, flowing out of a reservoir, basin, treatment plant, or industrial treatment plant, or part thereof.

elutriate -
To purify, separate, or remove by washing.

endogenous -
A diminished level of respiration in which materials previously stored by the cell are oxidized.

enzyme -
A protein that promotes a chemical reaction, enabling it to continue at body temperature.

filamentous bacteria -
These bacteria develop where carbohydrates are present and where there is low dissolved oxygen content. The result is bulking and poor settling. These organisms grow in a thread or filamentous form.

flights -
Wooden scrapers mounted on parallel chains to move sludge to a hopper at the end of a rectangular clarifier.

floc -
Small gelatinous masses formed in a liquid by the reaction of a coagulant added thereto, through biochemical processes, or by agglomeration.

flocculation -
The collection of coagulated suspended solids into a mass by gentle stirring.

flotation -
The raising of suspended matter to the surface of wastewater in a tank for removal by skimming.

fungi -
Small non-chlorophyll-bearing plants which lack roots, stems, or leaves, which occur (among other places) in water, wastewater, or wastewater effluents and grow best in the absence of light. Their decomposition after death may cause disagreeable tastes and odours in water; in some wastewater treatment processes they are helpful and in others they are detrimental.

Imhoff tank -
A wastewater treatment tank with two chambers for sedimentation and sludge digestion.

influent -
Water, wastewater, or other liquid flowing into a reservoir, basin, or treatment plant, or any unit thereof.

inorganic -
Chemical substances of mineral origin, or more correctly, not of basically carbon structure.

metabolize -
To perform the chemical changes in organic cells, providing energy for growth and activity.

microbes -
Microscopic organisms, especially pathogenic bacterium.

micro-organisms -
Minute organisms, either plant or animal, invisible or barely visible to the naked eye.

mixed liquor -
A mixture of activated sludge and organic matter undergoing treatment in the aeration tank.

nitrification -
(1) The conversion of nitrogenous matter into nitrates by bacteria.

(2) The treatment of a material with nitric acid.

nutrient -
Food for the growth of organisms.

organic -
Chemical substances of animal or vegetable origin, or more correctly, of basically carbon structure, comprising compounds consisting of hydrocarbons and their derivatives.

oxidation -
The act of combining with oxygen; any reaction which involves the loss of electrons from an atom.

oxygenation capacity -
In treatment processes, a measure of the ability of an aerator to supply oxygen to a liquid.

Parshall Flume -
A device used to measure liquid flow in a channel.

pH -
The measure of the acid/alkaline balance, expressed on a scale of 0 to 14, with 7 being neutral; 7 to 0 increasing acidity, and 7 to 14 increasing alkalinity.

preaeration -
A method of preparing wastewater for treatment by aeration to remove gases, add oxygen, float grease, etc.

protozoa -

Unicellular microscopic animals, protozoa consume bacteria, thus promoting the growth of new bacteria. They feed on the surface of biological floc and on dispersed bacteria, which results in a clear effluent. The presence of protozoa indicates that there is sufficient dissolved oxygen and a lack of toxic elements. There are two basic forms:

Free-swimming ciliate protozoa - Fine hairs allow these ciliates to swim rapidly. They have a high energy level and require a large quantity of organic food.

Stalked ciliate protozoa - Normally found in high-rate systems in equal numbers with the free-swimming ciliates, they attach themselves by their stalks to solid particles.

putrescible -

(1) The relative tendency of organic matter to undergo decomposition in the absence of oxygen.

(2) The susceptibility of wastewaters, effluent, or sludge to putrefaction.

(3) In water or wastewater analysis, the stability of a polluted water or raw or partially treated wastewater.

retention time (or period) -

The theoretical time required to displace the contents of a tank or unit at a given rate of discharge (volume divided by rate of feed). Also called detention time.

rotifers -

Multicellular microscopic animals which feed on bacteria and protozoa, rotifers exist only in the presence of dissolved oxygen and are an indication of a high degree of treatment. They are normally found in extended aeration systems.

stabilize -

To convert to a form that resists change. Organic material is stabilized by bacteria which convert the material to gases and other relatively inert substances. Stabilized organic material generally will not give off obnoxious odours.

sedimentation -

Settling or clarification; the process of allowing solids in water and sewage to sink to the bottom for easy removal.

supernatant -

The liquid standing above a sediment or precipitate.

suspended solids -

(1) Solids that either float on the surface of, or are in suspension in, water, wastewater, or other liquids, and which are largely removable by laboratory filtering.

(2) The quantity of material removed from wastewater in a laboratory test, as prescribed in "Standard Methods for the Examination of Water and Wastewater" and referred to as non-filterable residue.

total solids -

The sum of dissolved and undissolved constituents in water or wastewater, usually stated in milligrams per litre.

turbidity -

A condition in water caused by suspended matter; murkiness.

volatile solids -

The quantity of solids in water, wastewater, or other liquids, lost on ignition of the dry solids at 550°C.

weir -

A dam or enclosure in water or wastewater used to raise the water level or change the direction of its flow; with notches or a crest, it measures the flow.